# 2002 Performance Report
# General Chemistry and Microbiology Section

May 2003


Ministry of the Environment

# 2002 PERFORMANCE REPORT

# GENERAL CHEMISTRY AND MICROBIOLOGY SECTION

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## INTRODUCTION

The General Chemistry and Microbiology Section (GCMS) is part of the Ministry of the Environment's Laboratory Services Branch. The section is comprised of two units, the Water Chemistry and Microbiology Unit. The Water Chemistry Unit identifies and provides quantitative analysis for major ions, nutrients, and physical properties in a variety of matrices. The Microbiology Unit identifies and enumerates indicator bacteria of water and waste waters.

This report provides a brief outline of the analytical quality control (QC) program associated with sample analysis and examines 2002 performance data for each test in the Water Chemistry and Microbiology Units. GCMS strives to maintain a high standard of analytical performance through its quality assurance program. QC is an integral part of this process.

A number of changes have taken place in the General Chemistry and Microbiology Section since the 2001 performance report was issued. The following describes those changes.

**METHODS ADDED BY GCMS in 2002.**

Sulphide (E3100)

**METHODS DISCONTINUED BY GCMS in 2002.**

Acidity, Gran (E3248)
Acidity, Total Fixed Endpoint (E3248)
pH (E3248)
Fluoride (E3369)

## Table of Contents

## 1.0 PERFORMANCE REPORT FORMAT

The parameters are those analysed by the GCMS for 2002.

The performance report is organized alphabetically according to test name (eg. Dissolved Organic Carbon is filed under the heading "Carbon, Dissolved Organic") and second, by the method reference number. Detailed information concerning the format of each page is outlined below:

### 1.1 TEST DESCRIPTION

TITLE: The name of the test parameter.

IDENTIFICATION:

Laboratory — Location where the test is performed.

Method Reference No: — A number assigned by the Quality Management Unit to an analytical test method eg.(E3370).

Product Code: — LIMS code for analysis request.

Sample Type/Matrix: — The various sample types that can be routed to the method.

Method Introduced: — Date that the method was implemented at the laboratory.

Reporting Units: — Unit of measurement in which the results are reported.

Supervisor: — Name of supervisor/manger responsible for the method.

SAMPLING:
The type of container and preservative (if applicable) that is used and minimum volume of sample that is usually required. Any sample preparation that is normally performed in the field, is also indicated (1).

SAMPLE PREPARATION:
Sample preparation techniques which are usually performed at the laboratory before analysis.

ANALYTICAL PROCEDURE:
Brief summary of the analytical method used to determine the parameter.

INSTRUMENTATION:
Type of instrumentation used to perform the test. Examples: Automated continuous flow systems consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and readout system. Microcomputers are used to control the operation of analytical equipment and/or data acquisition.

REPORTING:
W and T are low level data qualifiers (2). A value reported as <=W is interpreted as not present , the value accompanying the remark is the lowest reportable value of the method under routine operating conditions. A value (multiple of W) reported as <=WE is interpreted as above for <=W following non-routine dilution of the sample to allow analysis of the target substance. A value reported as <T is interpreted as target substance identified, use caution in interpretation unless more sample data supports this result. A value reported as <TE is interpreted as above for <T following non routine dilution of the sample to allow analysis for the target substance.

To provide a consistent LSB approach to data reporting, GCMS calculates W from the standard deviation of duplicates ($S_2$), near zero, by rounding down to the nearest 1,2 or 5 digit (4). T is five times W. The latest calculations, valid at date of publication for W and T values of all active methods, are contained in this report (APPENDIX A).

Data is reported to a maximum of three significant figures.

CALIBRATION:
The number of standards used to calibrate the analytical system plus blanks if applicable.

CONTROLS:
The calibration, drift, recovery, and interference controls that are used when applicable to ensure that the system is operating properly.

MODIFICATIONS:
Modifications made to the test in 2002.

NOTES:
Explanatory notes which may aid the data user in interpreting results and information.

## 1.2 PERFORMANCE DATA SUMMARY

QUALITY CONTROL DATA FROM/TO: (Optional)
The period of time over which data were collected.

ANALYTICAL RANGE AND REPORTING UNIT:
The full scale value for the analytical range is given in concentration units.

CALIBRATION CONTROL:
Calibration control includes a table outlining the number of data collected over the selected time period, expected concentrations of the control standards, the calculated mean concentration of these standards, mean bias (mean concentration minus the expected concentration), and standard deviations of each control standard. The between run standard deviation (S), the within run standard deviation ($S_w$), the ratio $S/S_w$, and the historical control limits for standards sums and differences are provided.

RECOVERIES (Where applicable):

DUPLICATES:
The table outlines within run duplicate data collected over the selected time period. Data are sorted into a number of concentration spans. The standard deviation for duplicates is provided for each range. The coefficient of variation (%) is determined by dividing the mean standard deviation ($S_2$) for a particular concentration span by the mean concentration of duplicate results in that span and multiplying by 100.

OTHER CHECKS (Where applicable):
The table outlines the number of data collected over a selected time period, the calculated mean concentration of ie., blank, and standard deviation.

## 1.3 QUALITY CONTROL GRAPHICS

CALIBRATION CONTROL:
Calibration control standards sums and differences are plotted on a horizontal scale for the period of data collection (referred to on the graphs as "QUALITY CONTROL STANDARD A+B" for example). The vertical scale consists of the warning/control limits expressed on either side of the expected value. These limits were chosen from analytical performance data.

**NOTE:**

DATE FORMAT:
mm/dd/yy

## 2.0 ANALYTICAL QUALITY CONTROL PROGRAM

Quality control is a continuous process that involves constant checks of sample processing procedures. This report summarizes the QC data collected during analytical processing to monitor performance of the analytical system.

**Calibration Standards** are verified for identity, purity and concentration accuracy by comparison against independent sources wherever possible. A series of calibration standards are analyzed covering the analytical range.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, quality control may be used to evaluate: calibration, blank, recovery, sensitivity, potential interference, and sample repeatability.

Calibration and Blank

Calibration is controlled by a minimum of two quality control standards and a long term blank which are prepared and maintained independently of the calibration standards. The system is not calibrated with the quality control standards. The long term blank is Pure De-ionized Water (Pure-DW)used to prepare the quality control standards and has zero concentration of the target analyte. Control standards are prepared less frequently than calibration standards and errors in newly prepared calibration standards can be detected by this cross check. Newly prepared control standards are run in parallel with in-use control standards and must meet control requirements over three consecutive runs before the new standards are accepted for routine use.

The standard deviation of the control standards is used to estimate the between-run standard deviation (S) and is compared against the within-run standard deviation ($S_w$). If the ratio $S/S_w$ exceeds 1.5 then poor control of systematic error can be inferred (3). Values for S and $S_w$ are calculated as follows:

$$2S^2 = (S_A)^2 + (S_B)^2 \qquad 2S_w^2 = (S_{A-B})^2$$

Where

$S_A$ = standard deviation (s.d.)of control standard A
$S_B$ = s.d. of control standard B
$S_{A-B}$ = s.d. of the difference between control standards A and B

NOTE: If a second range is employed for a test, more control standards are used because, in many systems, the between-run standard deviations are concentration dependent.

Detailed description of the quality control processes are outlined in several LSB documents (2)(4)(5) and (6).

Control/Warning Limits

The control standards data are assessed and compared against the control/warning limits established from previous data to determine whether the calibration process is in control. The limits are set up initially based on method performance(4), and are reviewed when method and/or performance data reviews are conducted to determine if modifications are required based on historical data calculations. Control limits are calculated for the sums and differences of control standards (A,B,C,D) by the equations:

(A+B) $\pm 4(S_{A-B})$ for the sum of A+B
(B+C) $\pm 4(S_{B-C})$ for the sum of B+C
(C+D) $\pm 4(S_{C-D})$ for the sum of C+D

(A-B) $\pm 3(S_{A-B})$ for the difference of A-B
(B-C) $\pm 3(S_{B-C})$ for the difference of B-C
(C-D) $\pm 3(S_{C-D})$ for the difference of C-D

Note: Warning Limits are calculated by the same formulae above (using ±2 instead of 4 and 3 respectively).

If a control limit is exceeded, the analysis is stopped, corrective action taken and the control standards are re-analysed.

Recovery

Some methods require sample pre-treatment, such as digestion or extraction. A recovery check, suitable to that method, is required to estimate the efficiency of the pre-treatment. Recovery standards are usually prepared at 0%, 20% and 80% of full scale. The solutions are analysed in the same manner as routine samples. Although these solutions are not used to calibrate the instrument andcorrections for the blank are calculated and applied if necessary. For an analytical run to be accepted, the recoveries should be within ±(5% +T/2) of their expected values. (See Section 1.1 "Reporting" for T determination). The average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

Sensitivity and Baseline

Any change in the sensitivity of the instrumentation is monitored periodically during the run ,as defined by the method, by analysing a standard that is usually 80% of full scale, and comparing the reading to the original calibration standards. Baseline drift is usually recorded by periodic analysis, as defined by the method, of Pure-DW which does not contain any of the analyte, but may be treated to correspond to sample pre-treatment.

Interference

The interference check is run on any test where a substance may be present in concentrations that affect the results. The check is carried out near the threshold concentration of the interfering substance, beyond which the methodological safeguards used to minimize the interference are no longer effective. The check indicates that the interference has no effect up to the specified concentrations.

Sample Repeatability

Generally, one sample out of twenty is analysed in duplicate up to a maximum of three duplicates per analytical run. The samples are selected for non-adjacent, within-run duplicate analyses. By analysing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within an analytical run, can be determined. For results to be acceptable, at least two of the three duplicate pairs must conform to limits that are set based on historical performance.

Duplicate data are accumulated and usually sorted into 3 ranges of 0-10 or 0-20, 21-50, 51-100 percent of full scale. More ranges may be added where the analytical scale spans are greater than 2 log scales. When less than 3 data pairs are collected, the remark N.A. ( not available ) is reported. A standard deviation is calculated for each concentration range. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev. (1)*

$$S_1 = \sqrt{\frac{\sum_{i=1}^{n} (\bar{x} - x_i)^2}{n-1}}$$

Std. Dev. of Duplicates (2)*

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n'} (x_1 - x_2)_i^2}{2n'}}$$

* Standard deviations used for the data summaries.

Where $S_1$ = sample standard deviation
$S_2$ = duplicate difference standard deviation
n = number of data
$\bar{x}$ = mean of data
$x_i$ = $i^{th}$ result
$(x_1 - x_2)_i$ = difference of the $i^{th}$ duplicate
n' = number of duplicate pairs

The standard deviation ($S_2$) of the duplicate difference is also expressed as the coefficient of variation (CV).

$$CV = \frac{S_2}{\bar{X}} \times 100$$

## 2.1 PERFORMANCE SUMMARIES

# ALKALINITY, TOTAL FIXED ENDPOINT

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 09/07/80 |
| Method Reference No. | E3218 | Reporting Unit | mg/L as $CaCO_3$ |
| LIMS Product Code | PHALCO3218 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or Plastic |

## ANALYTICAL PROCEDURE:

Samples (20.0 mL) are titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, and conductivity are determined simultaneously.

## INSTRUMENTATION:

Automated modular titration system with computer control and data processing software.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |

## CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

## CONTROLS:

| | |
|---|---|
| Calibration | BL plus 4 standards, e.g. QCA |
| Drift | In run standards throughout the run (tap water diluted to 50% V/V) |

## NOTES:

May '97 the W value was changed from 0.2 to 0.5 after a review of 2 years low level duplicate data '94-95.

## ALKALINITY, TOTAL FIXED ENDPOINT (E3218)

QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

Analytical Range: to 1000 mg/L as $CaCO_3$

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 74 | 250 | 251.39 | 1.39 | 1.7517 |
| B: | 74 | 100 | 100.45 | 0.45 | 0.9784 |
| C: | 74 | 25 | 24.997 | -0.003 | 0.5736 |
| D: | 74 | 2.5 | 2.41 | -0.09 | 0.1229 |
| A+B: | | 350 | 351.84 | 1.84 | 2.5326 |
| A-B: | | 150 | 150.94 | 0.94 | 1.2797 |
| B+C: | | 125 | 125.45 | 0.44 | 1.2084 |
| B-C: | | 75 | 75.45 | 0.45 | 1.0547 |
| C+D: | | 27.5 | 27.41 | -0.09 | 0.5793 |
| C-D: | | 22.5 | 22.59 | 0.09 | 0.5938 |

| | | | | |
|---|---|---|---|---|
| s.d.(AB) | S(between runs): 1.42 | Sw(within run): | 0.90 | S/Sw: 1.6 |
| s.d.(BC) | S(between runs): 0.80 | Sw(within run): | 0.75 | S/Sw: 1.1 |
| s.d.(CD) | S(between runs): 0.42 | Sw(within run): | 0.43 | S/Sw: 1.0 |

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 343 | - | 357 | for | A+B |
| 144.75 | - | 155.25 | for | A-B |
| 121.5 | - | 128.5 | for | B+C |
| 72.4 | - | 77.6 | for | B-C |
| 25.82 | - | 29.18 | for | C+D |
| 21.24 | - | 23.76 | for | C-D |

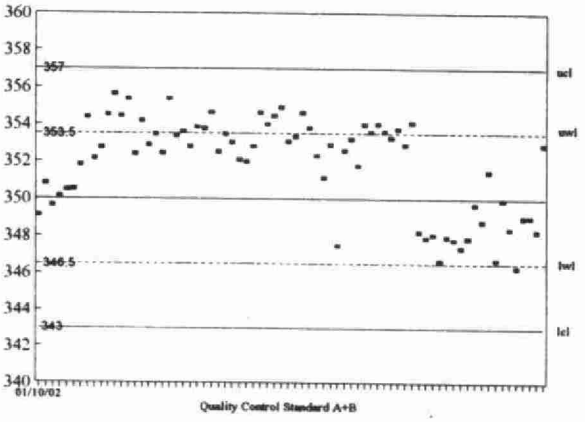
**DUPLICATES:**

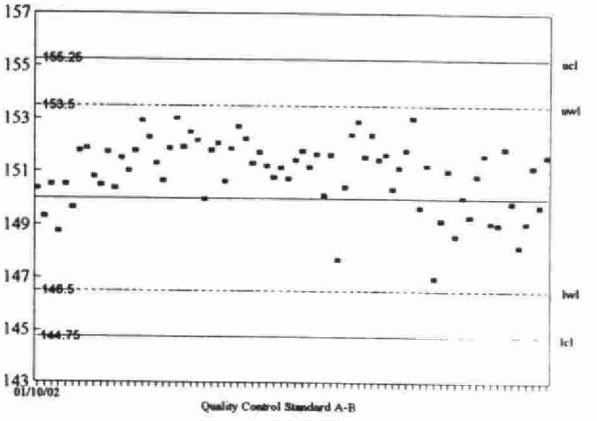
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 38 | 0 - 50 | 0.4241 | 1.9 |
| 46 | 51 - 100 | 0.4963 | 0.6 |
| 111 | 101 - 300 | 0.9276 | 0.5 |
| 16 | 301 - 1000 | 1.4211 | 0.3 |
| 211 | Overall | 0.8318 | |

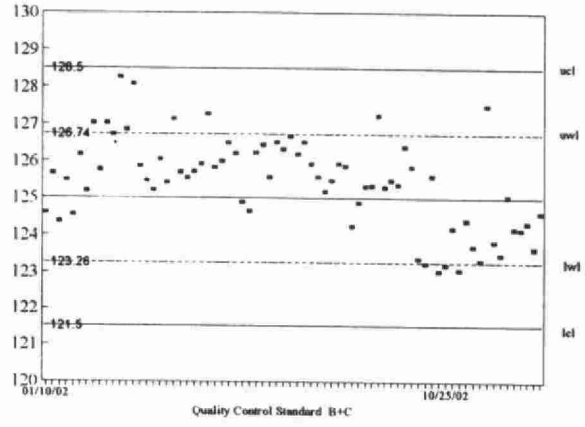
## ALKALINITY, TOTAL FIXED ENDPOINT (E3218)

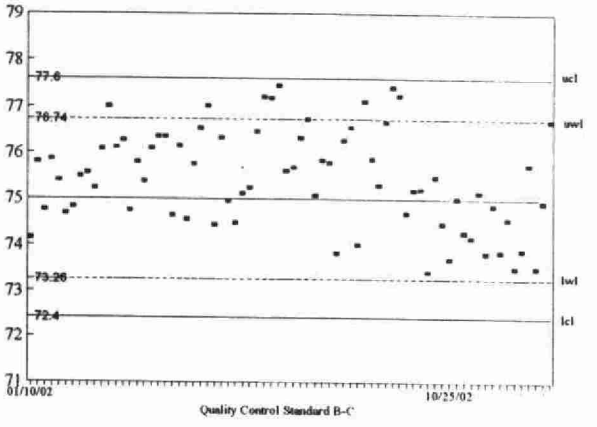
QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

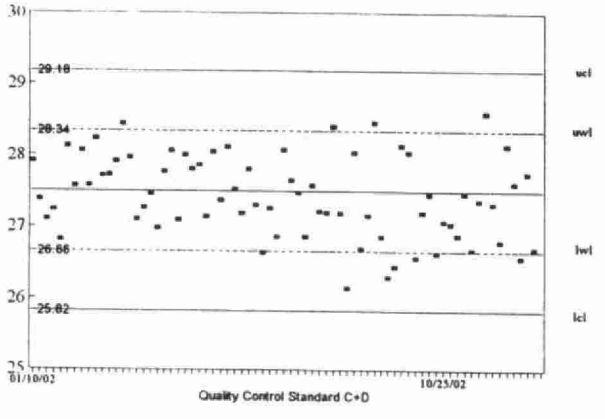
Analytical Range: to 1000 mg/L as $CaCO_3$

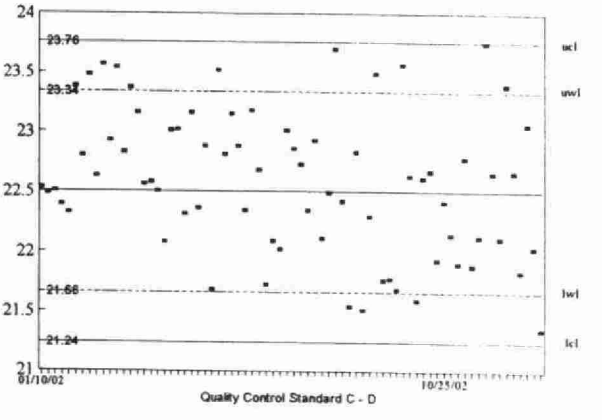


## CARBON, DISSOLVED INORGANIC

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3370 | Reporting Unit | mg/L as C |
| LIMS Product Code | DCSI3370 | Supervisor | P.Wilson |
| Sample Type/Matrix | Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.
Approximate absorbance: 0.3 at the full scale level.
Dissolved organic carbon, and reactive silicates are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system plus the following modules: air ($CO_2$-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g., QCA |
| Drift | BL , standard and BL every 10 samples |

**NOTES:**

December 1998: The HP data capture/processing system was replaced by Labtronics.

## Carbon; dissolved inorganic (E3370)

### Analytical Range: to 80 mg/L as C

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| A | 48 | 64 | 64.001 | 0.001 | 0.503 |
| B | 48 | 16 | 16.008 | 0.008 | 0.365 |
| C | 48 | 4 | 4.055 | 0.055 | 0.167 |
| A + B | | 80 | 80.009 | 0.009 | 0.648 |
| A - B | | 48 | 47.994 | -0.006 | 0.593 |
| B + C | | 20 | 20.063 | 0.063 | 0.494 |
| B - C | | 12 | 11.953 | 0.047 | 0.28 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.4391 |
| | Within Runs | 0.4193 |
| | Between/Within | 1.0472 |
| s.d.(BC) | Between Runs | 0.2836 |
| | Within Runs | 0.198 |
| | Between/Within | 1.4323 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 81.07 | 78.93 | 82.14 | 77.86 |
| A - B | 49.07 | 46.93 | 49.6 | 46.4 |
| B + C | 20.58 | 19.42 | 21.17 | 18.83 |
| B - C | 12.58 | 11.42 | 12.88 | 11.12 |

**DUPLICATES:**

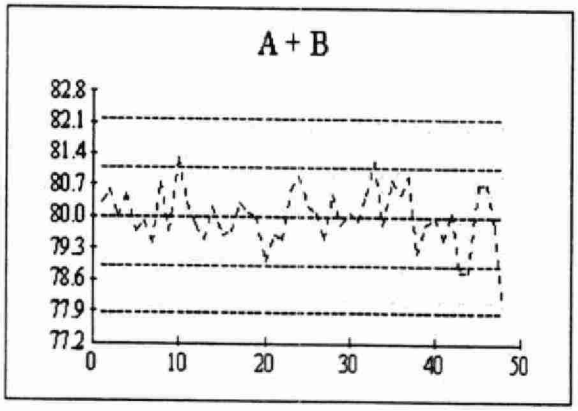
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 19 | 0 - 10% | 0.306 | 8.5 |
| 23 | 10 - 20% | 0.262 | 2.2 |
| 80 | 20 - 50% | 0.41 | 1.7 |
| 18 | 50 - 100% | 0.54 | 1.1 |
| 140 | Total | 0.397 | |

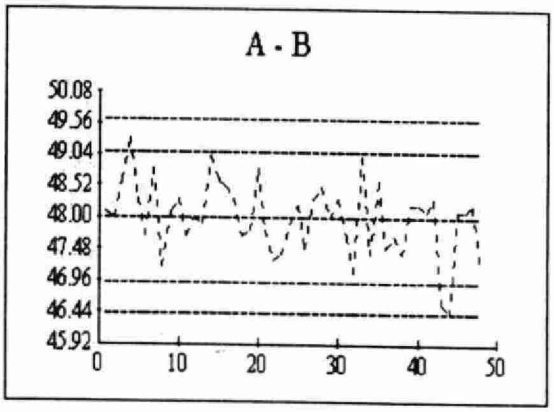
**OTHER CHECKS:**

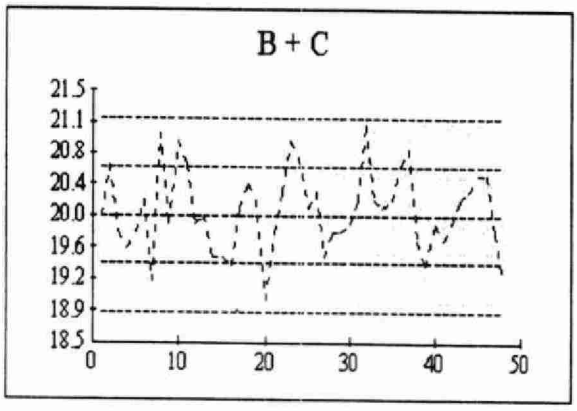
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 48 | 0.115 | 0.257 |

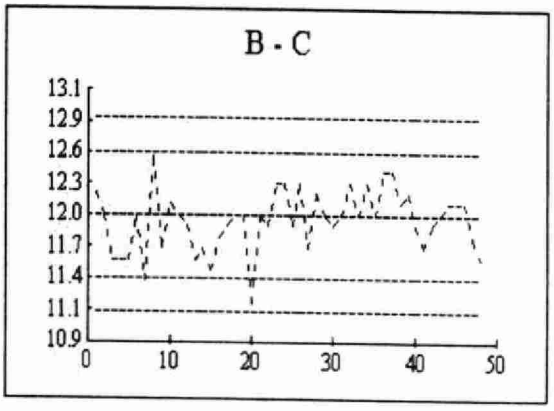
Carbon; dissolved inorganic ( E3370A )

QC Data; 1/1/02 to 12/31/02



## CARBON, DISSOLVED ORGANIC

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3370 | Reporting Unit | mg/L as C |
| LIMS Product Code | DCSI3370 | Supervisor | P.Wilson |
| Sample Type/Matrix | Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light (UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample. Approximate absorbance: 0.3 at the full scale level. Dissolved inorganic carbon, and reactive silicates are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system plus the following modules: nitrogen and air ($CO_2$ -free) supplies with flow controls, dialysis unit, UV digestor. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.1 | Current T value: 0.5 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g., QCA |
| Drift | BL , standard and BL every 10 samples |

**NOTES:**

December 1998: The HP data capture/processing system was replaced by Labtronics.

**Carbon; dissolved organic (E3370)**

**Analytical Range: to 20 mg/L as C**

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 48 | 16 | 15.966 | -0.034 | 0.137 |
| **B** | 48 | 4 | 3.973 | -0.027 | 0.148 |
| **C** | 48 | 1 | 0.985 | -0.015 | 0.13 |
| **A + B** | | 20 | 19.939 | -0.061 | 0.202 |
| **A - B** | | 12 | 11.993 | -0.007 | 0.202 |
| **B + C** | | 5 | 4.958 | -0.042 | 0.244 |
| **B - C** | | 3 | 2.988 | -0.012 | 0.136 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.1429 |
| | Within Runs | 0.1428 |
| | Between/Within | 1.0007 |
| s.d.(BC) | Between Runs | 0.1397 |
| | Within Runs | 0.0962 |
| | Between/Within | 1.4522 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 20.28 | 19.72 | 20.56 | 19.44 |
| A - B | 12.28 | 11.72 | 12.42 | 11.58 |
| B + C | 5.22 | 4.78 | 5.44 | 4.56 |
| B - C | 3.22 | 2.78 | 3.33 | 2.67 |

**DUPLICATES:**

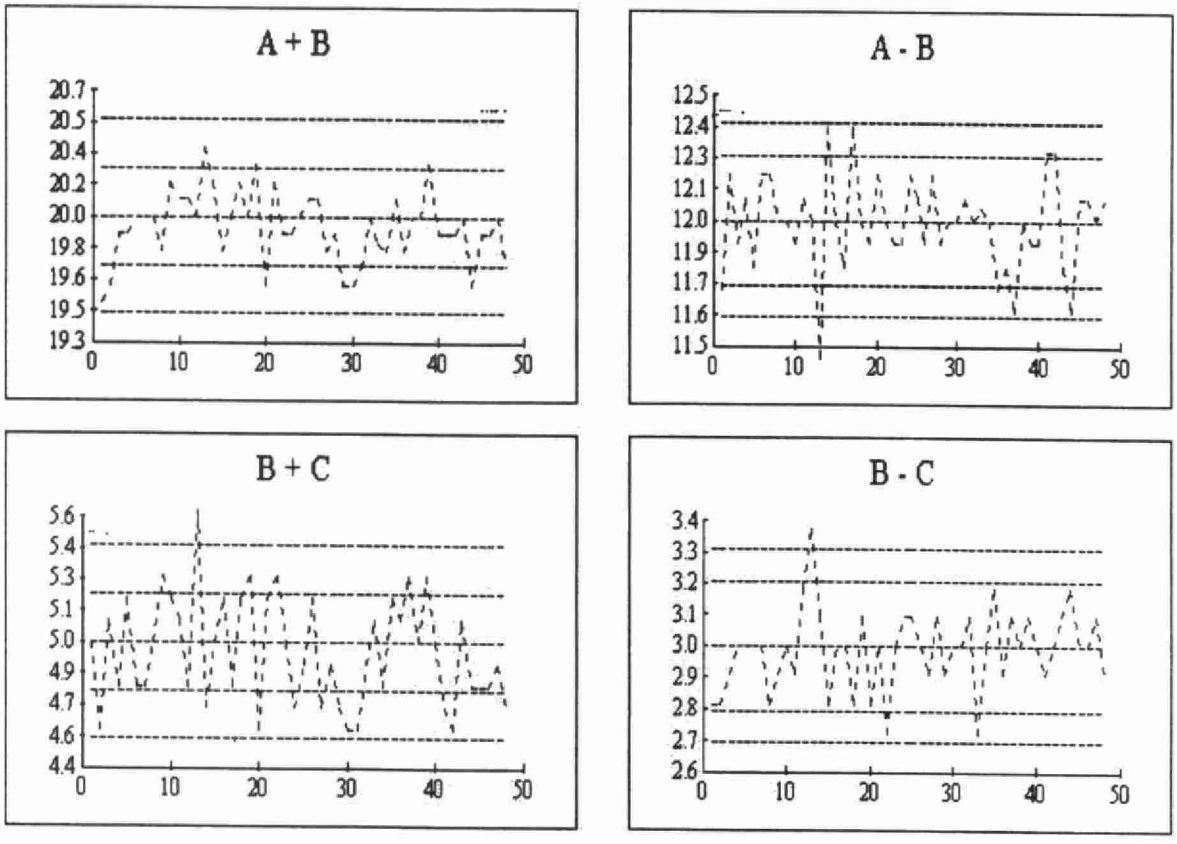
| Number | Conc. span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 62 | 0 - 10% | 0.104 | 7.4 |
| 37 | 10 - 20% | 0.145 | 4.9 |
| 40 | 20 - 50% | 0.134 | 2.4 |
| 3 | 50 - 100% | 0.058 | 0.4 |
| 142 | Total | 0.124 | |

**OTHER CHECKS:**

| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 48 | -0.004 | 0.143 |

Carbon; dissolved organic ( E3370A )

QC Data; 1/1/02 to 12/31/02




Note: For explanation of any exceedence, refer to raw data file.

# CHLORIDE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3004 | Reporting Unit | $\mu g/m^3$ as Cl |
| LIMS Product Code | ANION3004 | Supervisor | P. Wilson |
| Sample Type/Matrix | Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 3/4" or 1.9cm strip from 8"x10" filter |
| Container | 50 mL polypropylene tube |

## SAMPLING PREPARATION:

A 3/4" filter strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

## ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride ( mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation. The result is reported as $\mu g/m^3$ as Cl.
Nitrate and sulphate are determined simultaneously.

## INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.1 $\mu g/m^3$ | Current T value: 0.5 $\mu g/m^3$ |

## CALIBRATION:

6 standards

## CONTROLS:

| | |
|---|---|
| Calibration | MB, IS(n), CS1, and CS2 |
| Drift | Duplicate plus 2 standards approximately every 20 samples |
| Recovery | CS3 & CS4 |

**NOTES:**

To convert unit from mg/L to $\mu g/m^3$, the final concentration of Cl in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu g/m^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

**CHLORIDE** (E3004)

QUALITY CONTROL DATA FOR 01/17/02 TO 12/31/02

Analytical Range: to 28.61 µg/m$^3$

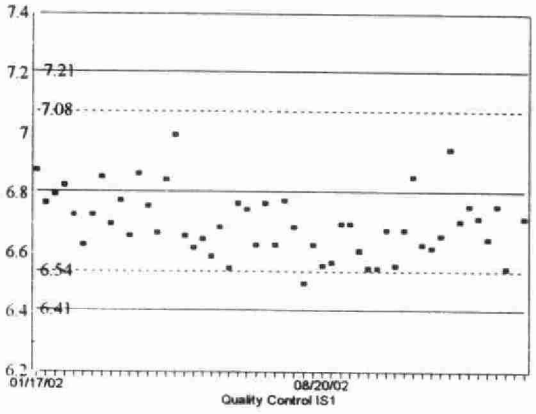
**DUPLICATES:**

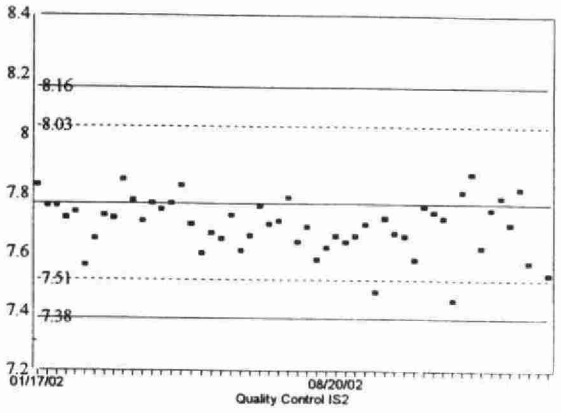
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 75 | 0.0 - 2.86 | 0.0392 | 14.6 |
| 1 | 2.89 - 7.15 | N.A. | N.A. |
| 0 | 7.18 - 14.31 | N.A. | N.A |
| 0 | 14.33 - 28.61 | N.A. | N.A. |
| 76 | Overall | 0.0397 | |

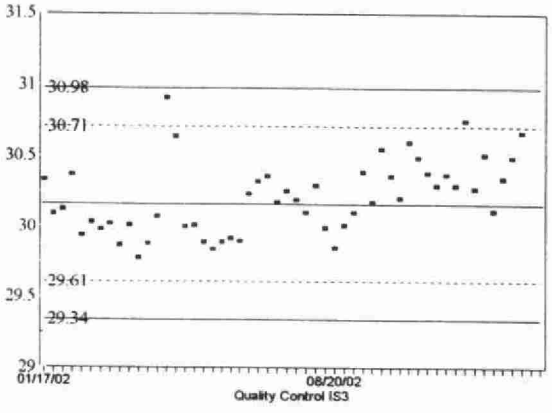
## CHLORIDE (E3004)

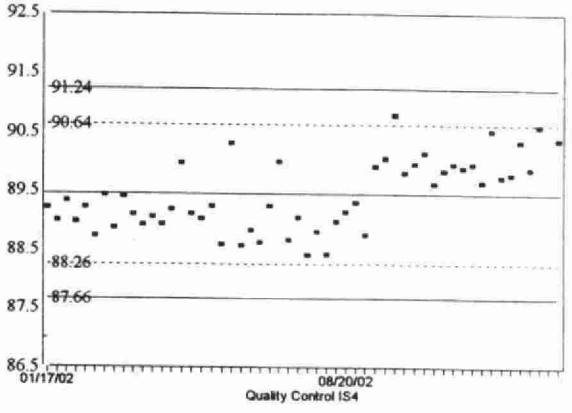
QUALITY CONTROL DATA FROM 01/17/02 TO 12/31/02

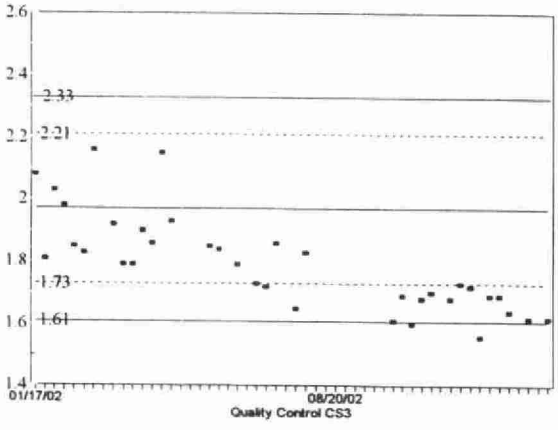
Analytical Range For IS Controls: to 100 mg/L
Analytical Range For CS Controls: to 28.61 µg/m$^3$

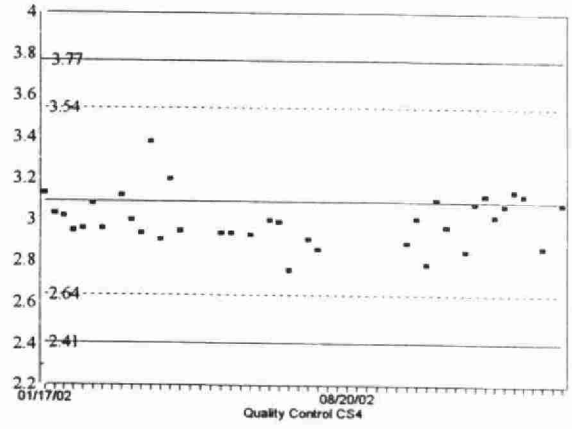



Note: For explanation of any exceedence, refer to raw data file.

# CHLORIDE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 31412 |
| Method Reference No. | E3013 | Reporting Unit | µg/g as Cl |
| LIMS Product Code | ANION3013, CL3013 | Supervisor | P. Wilson |
| Sample Type/Matrix | Soil and Sediment | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 20 g |
| Container | glass or plastic |

**SAMPLING PREPARATION:**

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

**ANALYTICAL PROCEDURE:**

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride ( mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as Cl.
Sulphate is determined simultaneously.

**INSTRUMENTATION:**

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 µg/g | Current T value: 2.5 µg/g |

**CALIBRATION:**

8 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | MB, IS(n), CS1, and CS2 |
| Drift | Duplicate plus 2 standards approximately every 20 samples |

**CHLORIDE** (E3013)

QUALITY CONTROL DATA FOR 2002

Analytical Range: to 1000 µg/g

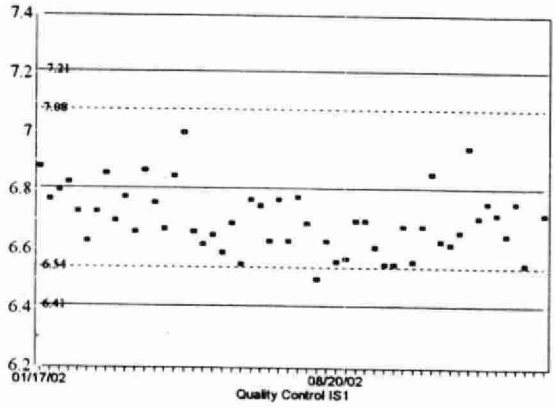
**DUPLICATES:**

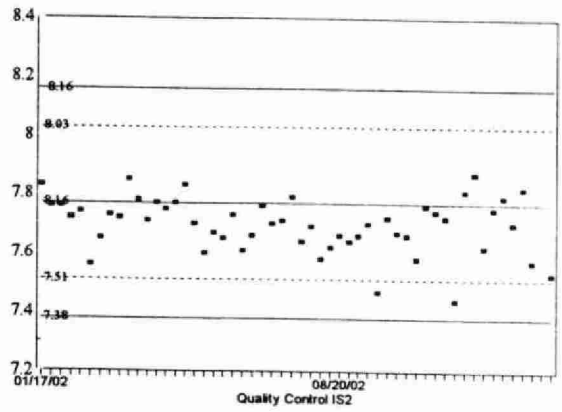
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 13 | 0.0 - 200 | 0.3913 | 0.7 |
| 1 | 201 - 500 | N.A. | N.A. |
| 0 | 500 - 1000 | N.A. | N.A. |
| 14 | Overall | 0.4500 | |

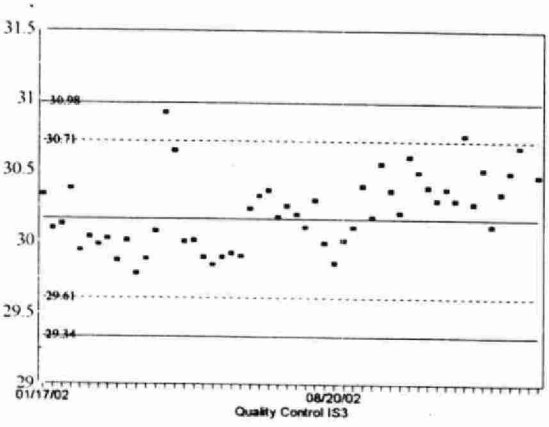
## CHLORIDE (E3013)

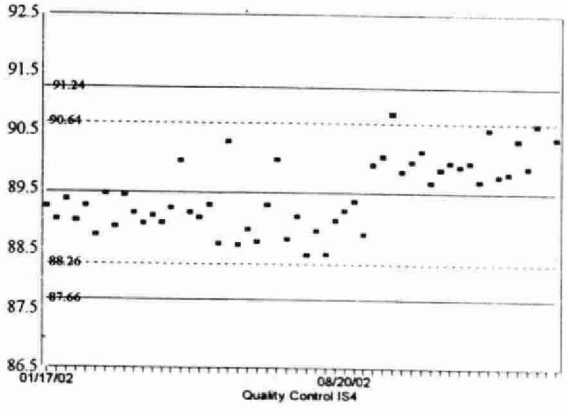
QUALITY CONTROL DATA FROM 01/17/02 TO 12/31/02

Analytical Range For IS Controls: to 100 mg/L



# CHLORIDE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/05/75 |
| Method Reference No. | E3016 | Reporting Unit | mg/L as Cl |
| LIMS Product Code | CL3016 | Supervisor | P. Wilson |
| Sample Type/Matrix | Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water, Ground Water, Leachate, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required: | 10 mL |
| Container: | Plastic |

## ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate (red), and the absorbance of the latter is measured colourimetrically.

Approximate absorbance: 0.5 at the full scale level.

## INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm light path at 480 nm.

Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |

## CALIBRATION:

BL plus 12 standards

## CONTROLS:

| | |
|---|---|
| Calibration: | LTBL plus 3 standards, e.g. QCA |
| Drift: | BL and standard after every 12 samples |

## NOTES:

April 1998: The HP data capture/processing system was replaced by Labtronics. Two additional Calibration standards were added at the low end of the curve.

## Chloride (E3016)

### Analytical Range: to 100 mg/L as Cl

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| A | 72 | 75 | 75.169 | 0.169 | 0.261 |
| B | 72 | 25 | 25.243 | 0.243 | 0.122 |
| C | 72 | 5 | 4.985 | -0.015 | 0.081 |
| A + B | | 100 | 100.411 | 0.411 | 0.332 |
| A - B | | 50 | 49.926 | -0.074 | 0.236 |
| B + C | | 30 | 30.228 | 0.228 | 0.164 |
| B - C | | 20 | 20.258 | 0.258 | 0.126 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.2035 |
| | Within Runs | 0.1669 |
| | Between/Within | 1.2193 |
| s.d.(BC) | Between Runs | 0.1037 |
| | Within Runs | 0.0891 |
| | Between/Within | 1.1639 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 100.7 | 99.3 | 101.3 | 98.7 |
| A - B | 50.7 | 49.3 | 51 | 49 |
| B + C | 30.34 | 29.66 | 30.7 | 29.3 |
| B - C | 20.34 | 19.66 | 20.5 | 19.5 |

**DUPLICATES:**

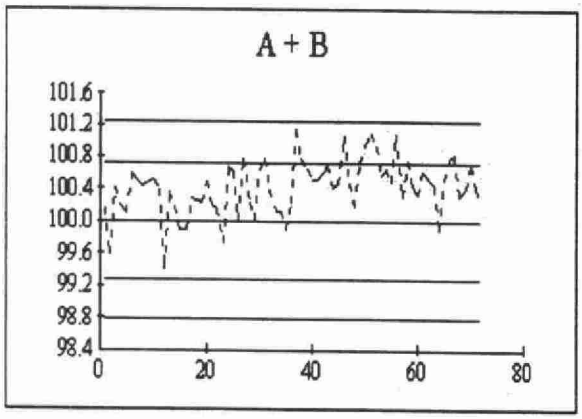
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 43 | 0 - 10% | 0.092 | 1.7 |
| 55 | 10 - 20% | 0.168 | 1.2 |
| 71 | 20 - 50% | 0.212 | 0.7 |
| 24 | 50 - 100% | 0.552 | 0.8 |
| 193 | Total | 0.254 | |

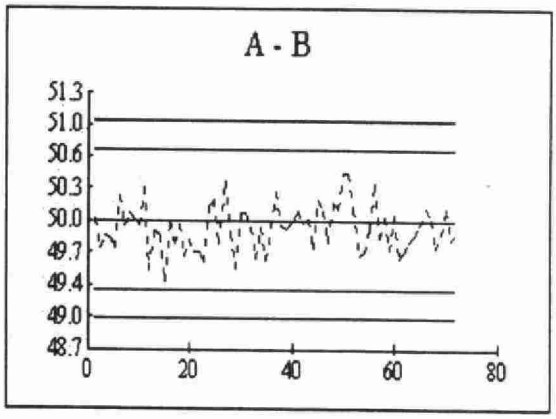
**OTHER CHECKS:**

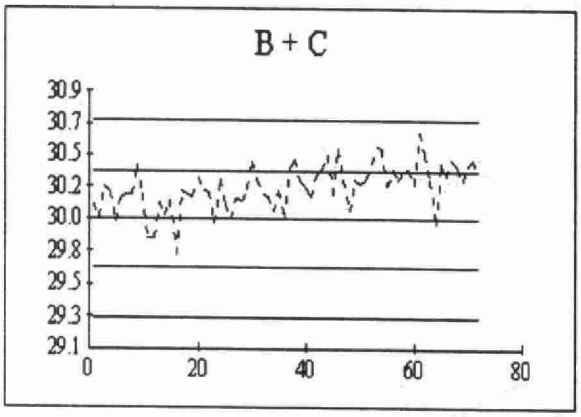
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 72 | 0.035 | 0.074 |

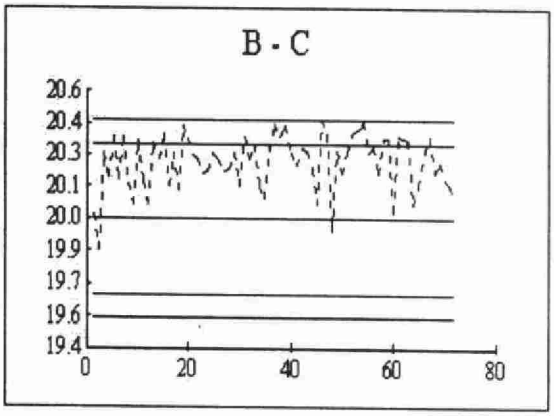
Chloride ( E3016A )

QC Data; 1/1/02 to 12/31/02



# CHLOROPHYLL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/75 |
| Method Reference No | E3169 | Reporting Unit | µg/L |
| LIMS Product Code | CHL3169 | Supervisor | P. Wilson |
| Sample Type/Matrix | Effluent, Drinking Water, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 1000 mL for clear samples; 500 mL if visibly green |
| Container | Glass or plastic |
| Other | In the field a sample is filtered through a nylon filter. The filter is folded and then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish labelled with the sample number and sample volume filtered, the dish is kept in the dark or wrapped in aluminum foil, and shipped immediately, or kept frozen. |

## ANALYTICAL PROCEDURE:

Chlorophyll 'a', chlorophyll 'b', and corrected chlorophyll 'a' (for pheophytin 'a') are determined by the extraction of the pheopigments into an acetone-water solvent followed by two computer controlled spectrophotometric scans with measurements at 630, 645 and 663 (665 for acidified) nm absorbance measurements. Also, the minimum absorbance between 710 and 750 is measured to allow for a correction due to turbidity. SCOR-UNESCO equations are used for all chlorophyll calculations.

## INSTRUMENTATION:

-Automated modular continuous flow scanning spectrophotometer system
-Computer system for control of sampling, timing and data processing (i.e. data capture, calculations and transfer of results to LIMS)

## REPORTING:

| | | | |
|---|---|---|---|
| Chlorophyll a; corrected<br>Chlorophyll a; total<br>Chlorophyll b; total | Maximum Significant Figures: 3 | Current W value: 1.0<br>Current W value: 0.2<br>Current W value: 0.1 | Current T value: 5.0<br>Current T value: 1.0<br>Current T value: 0.5 |

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 2 "standards", e.g.QCA |
| Drift | "standard",BL every 20 samples |

## NOTES:

"Standards" are prepared from chlorophyll "a" and "b", but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

**NOTES** (cont):

Jan 1999, the microcomputer system was replaced with a 486 PC The software written in PET BASIC was replaced by software written in CLIPPER.

May 2000 the Bechman DU7 spectrophotometer was replaced with a Bechman DU600 instrument.

## CHLOROPHYLL "a" (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 34 | 3.0 | 3.05 | 0.05 | 0.0992 |
| B: | 34 | 1.0 | 1.00 | 0.00 | 0.1093 |
| A+B: | | 4.0 | 4.06 | 0.06 | 0.1819 |
| A-B: | | 2.0 | 2.05 | 0.05 | 0.1024 |

s.d.(AB) S(between runs): 0.10 Sw(within run): 0.07 S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

**DUPLICATES:**

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 7 | 0 - 5.0 | 0.1230 | 10.7 |
| 5 | 5.1 - 10.0 | 0.7398 | 10.9 |
| 4 | 10.1 - 25.0 | 0.7550 | 4.6 |
| 16 | Overall | 0.5658 | |

Note: This table represents duplicate data from year 2001. No duplicate data for year 2002.

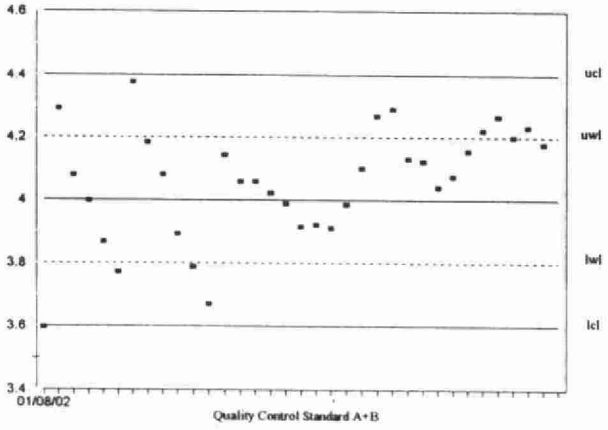
**OTHER CHECKS:**

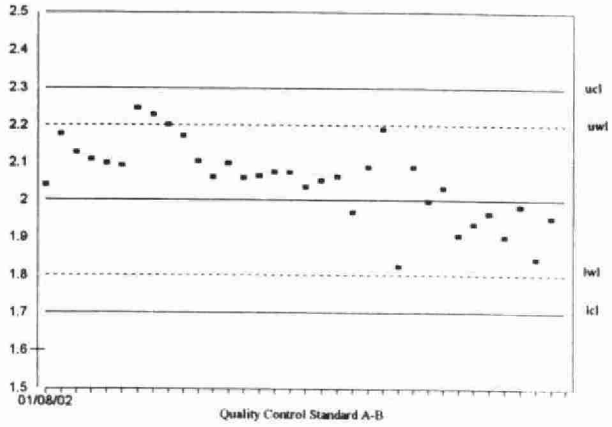
| | n | Mean | Standard Deviation (1) |
|---|---|---|---|
| Long Term Blank | 34 | 0.0103 | 0.0183 |
| Filtered Blank | 34 | 0.0095 | 0.0179 |

## CHLOROPHYLL "a" (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L




Note: For explanation of any exceedence, refer to raw data file.

## CHLOROPHYLL "a", ACIDIFIED (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 35 | 2.4 | 2.66 | 0.26 | 0.1237 |
| B: | 35 | 0.8 | 0.85 | 0.05 | 0.1169 |
| A+B: | | 3.2 | 3.51 | 0.31 | 0.2202 |
| A-B: | | 1.6 | 1.81 | 0.21 | 0.0971 |

s.d.(AB) S(between runs): 0.12 Sw(within run): 0.07 S/Sw: 1.75

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.4 - 4.0 for A+B
1.0 - 2.2 for A-B

**DUPLICATES:**

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 5 | -0.5 - 1.0 | 0.2268 | 35.2 |
| 0 | 1.1 - 2.0 | N.A. | N.A. |
| 2 | 2.1 - 5.0 | 0.0461 | 1.1 |
| 6 | 5.1 - 10.0 | 0.0604 | 7.9 |
| 0 | 10.1 - 100 | N.A. | N.A. |
| 14 | Overall | 0.4182 | |

Note: This table represents duplicate data from year 2001. No duplicate data for year 2002.

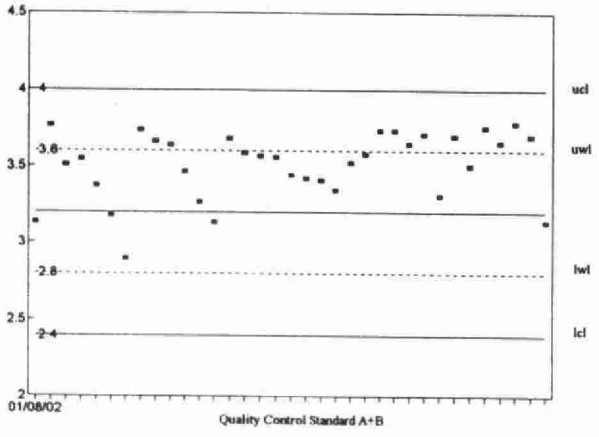
**OTHER CHECKS:**

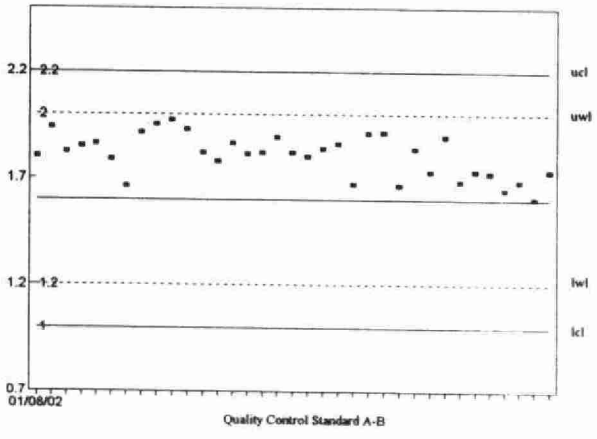
| | n | Mean | Standard Deviation (1) |
|---|---|---|---|
| Long Term Blank | 35 | -0.0009 | 0.0589 |
| Filtered Blank | 35 | -0.0034 | 0.0523 |

## CHLOROPHYLL "a", ACIDIFIED (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L



## CHLOROPHYLL "b" (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 35 | 3.0 | 2.92 | -0.08 | 0.0676 |
| B: | 35 | 1.0 | 1.00 | 0.00 | 0.0696 |
| A+B: | | 4.0 | 3.92 | -0.08 | 0.1128 |
| A-B: | | 2.0 | 1.92 | -0.08 | 0.0780 |

s.d.(AB) S(between runs):0.07 Sw(within run): 0.06 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

**DUPLICATES:**

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 5 | 0 - 1.0 | 0.2254 | 29.8 |
| 3 | 1.1 - 2.0 | 0.1224 | 8.0 |
| 2 | 2.1 - 5.0 | 0.0934 | 3.5 |
| 10 | Overall | 0.1779 | |

Note: This table represents duplicate data from year 2001. No duplicate data for year 2002.

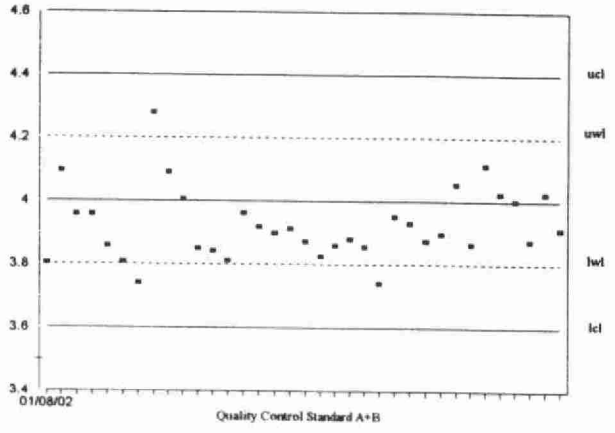
**OTHER CHECKS:**

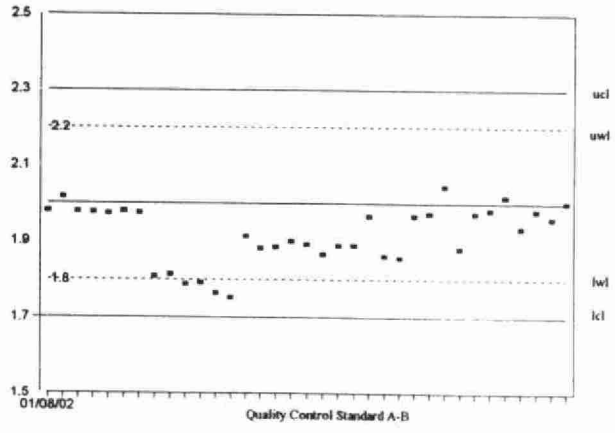
| | n | Mean | Standard Deviation (1) |
|---|---|---|---|
| Long Term Blank | 35 | 0.0139 | 0.0279 |
| Filtered Blank | 35 | 0.0118 | 0.0286 |

## CHLOROPHYLL "b" (E3169)

QUALITY CONTROL DATA FROM 01/08/02 TO 12/20/02

Reporting Unit: µg/L



# COLOUR, TRUE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 13/03/84 |
| Method Reference No. | E3219 | Reporting Unit | TCU |
| LIMS Product Code | COL3219 | Supervisor | P. Wilson |
| Sample Type/Matrix | Effluent, Industrial Waste, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.
Approximate absorbance: 0.3 at the full scale level.

## INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm). Turbidity measurement is through a 5.0 cm. light path using a different broadband filter (660-740 nm). Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |

## CALIBRATION:

BL plus 6 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift | BL and standard after every 10 samples |

## NOTES:

The HP data capture/processing system was replaced by Labtronics in November 1998.

## Colour; true (E3219)

## Analytical Range: to 100 TCU

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 44 | 70 | 70.826 | 0.826 | 0.517 |
| **B** | 44 | 25 | 25.543 | 0.543 | 0.425 |
| **C** | 44 | 7.5 | 7.239 | 0.261 | 0.335 |
| **A + B** | | 95 | 96.369 | 1.369 | 0.736 |
| **A - B** | | 45 | 45.283 | 0.283 | 0.595 |
| **B + C** | | 32.5 | 32.782 | 0.282 | 0.674 |
| **B - C** | | 17.5 | 18.304 | 0.804 | 0.362 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.4733 |
| | Within Runs | 0.4207 |
| | Between/Within | 1.125 |
| s.d.(BC) | Between Runs | 0.3823 |
| | Within Runs | 0.256 |
| | Between/Within | 1.4934 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 96.11 | 93.59 | 97.82 | 92.18 |
| A - B | 46.46 | 43.59 | 47.11 | 42.89 |
| B + C | 33.43 | 31.57 | 34.35 | 30.65 |
| B - C | 18.43 | 16.52 | 18.89 | 16.11 |

**DUPLICATES:**

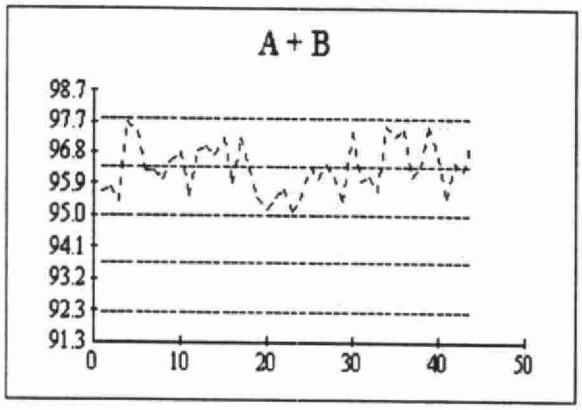
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 74 | 0 - 10% | 0.285 | 11.1 |
| 23 | 10 - 20% | 0.387 | 2.4 |
| 22 | 20 - 50% | 0.693 | 2.2 |
| 2 | 50 - 100% | N.A. | N.A. |
| 121 | Total | 0.418 | |

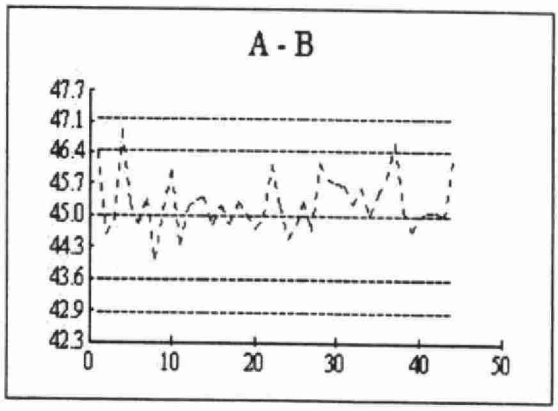
**OTHER CHECKS:**

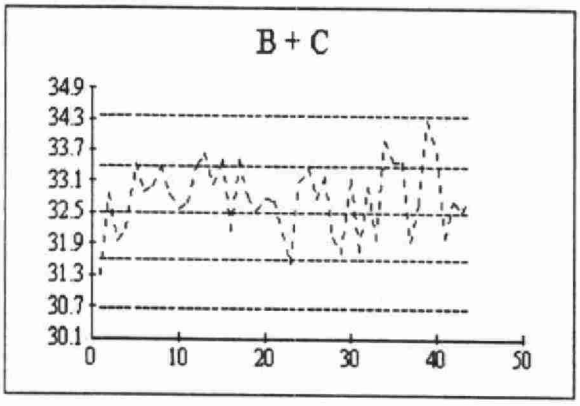
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 44 | -0.571 | 0.42 |

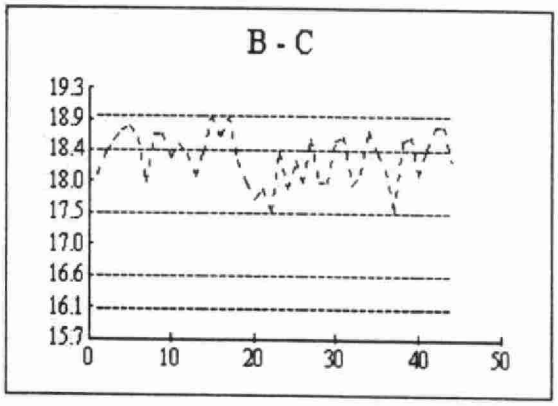
Colour;true [ E3219A ]

QC Data; 1/1/02 to 12/31/02



# CONDUCTIVITY

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced: | 01/04/74 |
| Method Reference No: | E3218 | Reporting Units: | µS/cm at 25°C |
| LIMS Product Code: | PHALCO3218,CONDPH3218 | Supervisor: | P. Wilson |
| Sample Type/Matrix: | Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required: | 50 mL |
| Container: | Glass or plastic |

## ANALYTICAL PROCEDURE:

After equilibration at room temperature, the conductivity of the sample is measured. Temperature compensation is applied by the system. Total fixed endpoint alkalinity and pH are determined simultaneously.

## INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler and conductivity meter with cell plus computer control and data processing software.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |

## CONTROLS:

| | |
|---|---|
| Calibration: | LTBL plus 4 standards, e.g. QCA |
| Drift: | In run standards throughout the run (tap water diluted to 50% V/V) |

## CONDUCTIVITY (E3218)

QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

Analytical Range: to 2000 µS/cm

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 75 | 1413 | 1412.88 | -0.12 | 3.6270 |
| B: | 75 | 718 | 718.74 | 0.74 | 2.1881 |
| C: | 75 | 147 | 148.22 | 1.23 | 0.7117 |
| D: | 75 | 37.1 | 37.41 | 0.31 | 0.7525 |
| A+B: | | 2131 | 2131.62 | 0.62 | 5.0174 |
| A-B: | | 695 | 694.13 | -0.87 | 3.2729 |
| B+C: | | 865 | 866.97 | 1.97 | 2.5142 |
| B-C: | | 571 | 570.51 | -0.49 | 2.0658 |
| C+D: | | 184.1 | 185.64 | 1.54 | 1.1284 |
| C-D: | | 109.9 | 110.82 | 0.92 | 0.9339 |

| | | | | |
|---|---|---|---|---|
| s.d.(AB) | S(between runs): 2.99 | Sw(within run): | 2.31 | S/Sw: 1.3 |
| s.d.(BC) | S(between runs): 1.63 | Sw(within run): | 1.46 | S/Sw: 1.1 |
| s.d.(CD) | S(between runs): 0.73 | Sw(within run): | 0.66 | S/Sw: 1.1 |

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 2109.8 | - | 2152.2 | for | A+B |
| 679.1 | - | 710.9 | for | A-B |
| 851.9 | - | 878.1 | for | B+C |
| 561.2 | - | 580.8 | for | B-C |
| 180.04 | - | 188.16 | for | C+D |
| 106.86 | - | 112.94 | for | C-D |

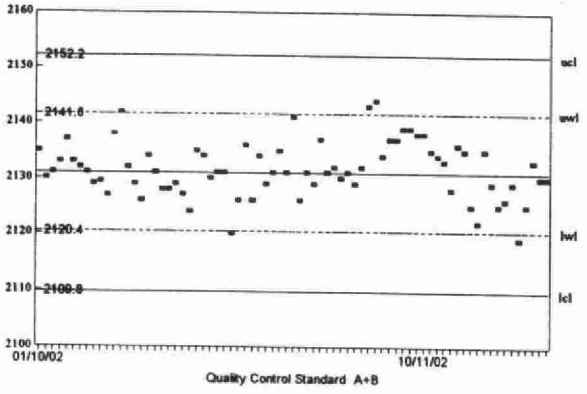
**DUPLICATES:**

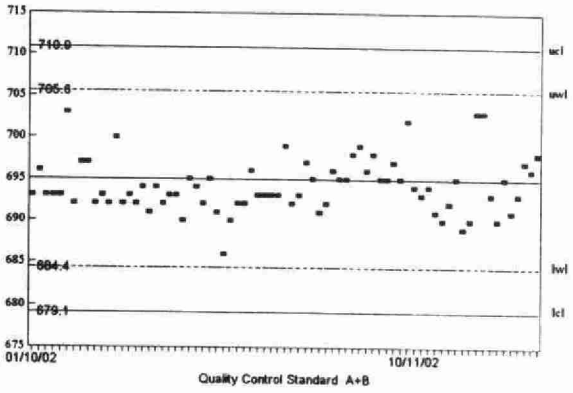
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 48 | 0 - 200 | 1.0714 | 1.1 |
| 68 | 201 - 400 | 2.1407 | 0.8 |
| 74 | 401 - 1000 | 2.5202 | 0.4 |
| 17 | 1001 - 2000 | 2.8851 | 0.2 |
| 10 | 2001 - 10000 | 3.8730 | 0.1 |
| 217 | Overall | 2.2802 | |

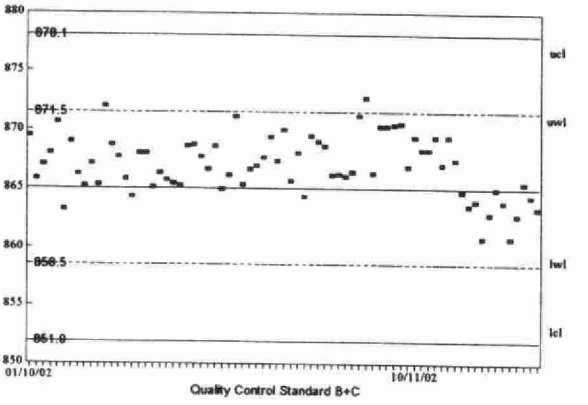
## CONDUCTIVITY (E3218)

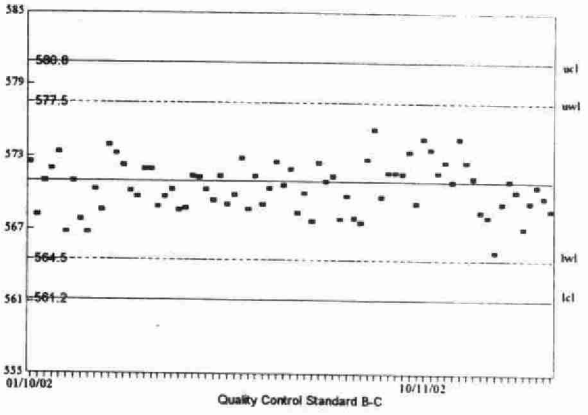
QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

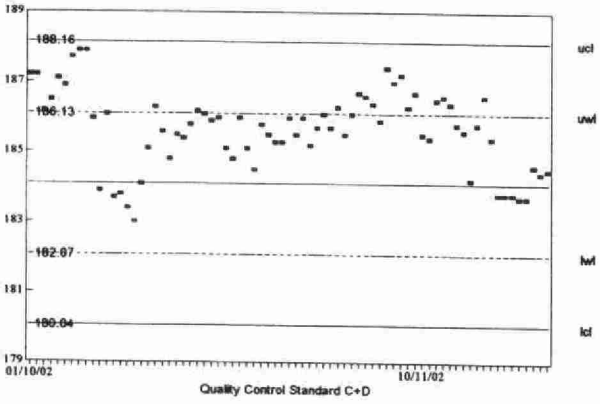
Analytical Range: to 2000 µS/cm

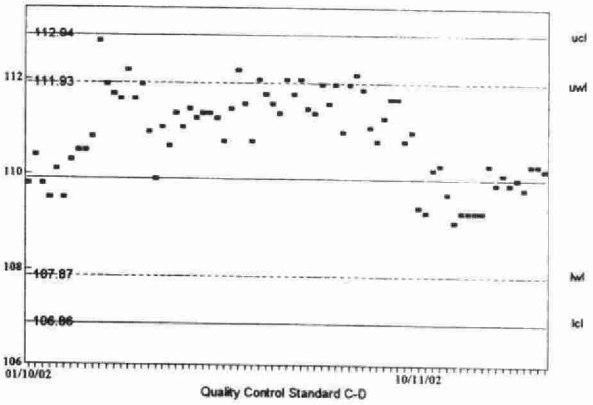


## CYANIDE, FREE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 35795 |
| Method Reference No. | E3015 | Reporting Unit | Aqueous: mg/L as $CN^-$<br>Solid: µg/g as $CN^-$ |
| LIMS Product Code | CNF3015 | Supervisor | P. Wilson |
| Sample Type/Matrix | **Aqueous:** Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent.<br>**Solid:** Sediment, Dried Sludge, Industrial Waste | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | **Aqueous**: 500 mL + 10 drops of 50% w/v NaOH **Solid** : 5 g, minimum |
| Container: | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Free cyanides are the simple and weakly dissociable cyanides which form HCN upon acidification to pH4.0 (such as HCN and KCN). The automated determination of free cyanide exposes the sample to distillation which isolates HCN under specific acidic conditions. A zinc sulphate solution is included which eliminates interference from complexed iron cyanides. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system by an autosampler.
Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours and then centrifuged. The supernatant is decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as µg/g wet and moisture content is reported by a separate method.

**INSTRUMENTATION:**

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm.
Skalar data capture and data processing software with computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.001 mg/L<br>0.01 µg/g | Current T value: 0.005mg/L<br>0.05 µg/g |

**CALIBRATION:**

BL plus 6 standards (S0 to S5)

**NOTE:**

December 2002, vegetation matrix removed.

**CONTROLS:**

| | |
|---|---|
| Calibration: | LTB plus 2 standards , e.g. QCA |
| Drift: | BL and check standards |

## CYANIDE, FREE (E3015)

QUALITY CONTROL DATA FROM 02/11/98 TO 10/16/02

Analytical Range: to 0.2 mg/L as CN

### CALIBRATION CONTROL:

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 55 | 0.15 | 0.1513 | 0.0013 | 0.0033 |
| B: | 55 | 0.02 | 0.0186 | -0.0014 | 0.0013 |
| A+B: | | 0.17 | 0.1699 | -0.0001 | 0.0035 |
| A-B: | | 0.13 | 0.1327 | 0.0027 | 0.0035 |

s.d.(AB) S(between runs): 0.0025 Sw(within run): 0.0024 S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B
0.118 - 0.142 for A-B

### REFERENCE MATERIAL:

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| KCN: | 55 | 0.10 | 0.0973 | -0.0027 | 0.0074 |
| FeCN:* | 20 | <0.001* | 0.0013 | 0.0003 | 0.0009 |

* 2000 to 2002 data

FeCN is not expected to be detected for free cyanide. Results should be <= w or <0.001 although standard tested is 0.10 mg/L.

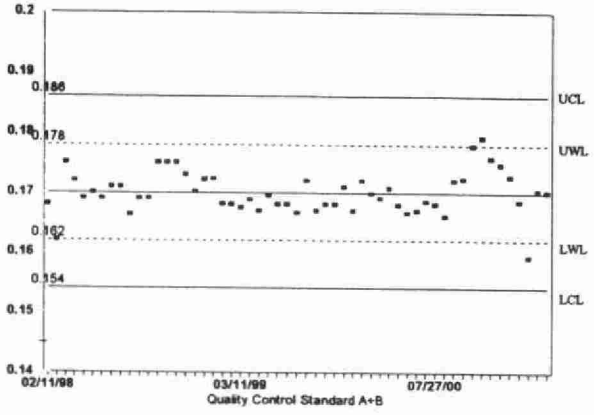
### DUPLICATES:

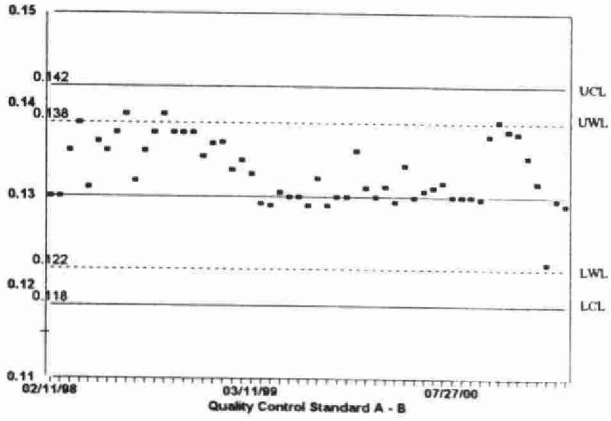
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 108 | 0 - 0.020 | 0.0005 | 14.9 |
| 3 | 0.021 - 0.040 | 0.0041 | 13.2 |
| 12 | 0.041 - 0.100 | 0.0030 | 4.6 |
| 10 | 0.101 - 0.200 | 0.0036 | 2.4 |
| 133 | Overall | 0.0015 | |

## **CYANIDE, FREE** (E3015)

QUALITY CONTROL DATA FROM 02/11/98 TO 10/16/02

Analytical Range: to 0.2 mg/L as CN



## CYANIDE, TOTAL

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 35795 |
| Method Reference No. | E3015 | Reporting Unit | Aqueous: mg/L as $CN^-$<br>Solid: µg/g as $CN^-$ |
| LIMS Product Code | CN3015, TCLPCN3015 | Supervisor | P. Wilson |
| Sample Type/Matrix | **Aqueous:** Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent.<br>**Solid:** Soil, Sediment, Dried Sludge, Industrial Waste | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | Aqueous: 500 mL + 10 drops of 50% w/v NaOH **Solid** : 5 g, minimum |
| Container: | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Total cyanides include free, simple (HCN,KCN) and weakly dissociable cyanides ($Ni(CN)_4$) as well as those complexed cyanides that decompose to form free cyanides that distill out as HCN in an acidic environment. The automated determination of total cyanide exposes the sample to ultraviolet radiation to break down organic metallic and alkali-complexed cyanide compounds to free cyanide. The distillation step isolates HCN under specific acidic conditions. The sequential combination of UV digestion plus distillation yields the measurement of "total cyanide" . Cyanide is measured colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system from an autosampler.
Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours, then centrifuged. The supernatant is then decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as µg/g wet and moisture content is reported by a separate method.

**INSTRUMENTATION:**

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm.
Skalar data capture and data processing software with computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.001 mg/L<br>0.01 µg/g | Current T value: 0.005mg/L<br>0.05 µg/g |

**CALIBRATION:**

BL plus 6 standards (S0 to S5)

**CONTROLS:**

| | |
|---|---|
| Calibration: | LTB plus 2 standards , e.g. QCA |
| Drift: | BL and check standards |

**NOTE:**

TCLPCN3015, LIMS product code was added on April 2001.

## CYANIDE, TOTAL (E3015)

QUALITY CONTROL DATA FROM 01/02/02 TO 12/13/02

Analytical Range: to 0.2 mg/L as CN for aqueous samples
Analytical Range: to 0.2 µg/g as CN for soil samples

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 35 | 0.15 | 0.1508 | 0.0008 | 0.0022 |
| B: | 35 | 0.02 | 0.0203 | 0.0003 | 0.0010 |
| A+B: | | 0.17 | 0.1711 | 0.0011 | 0.0029 |
| A-B: | | 0.13 | 0.1305 | 0.0005 | 0.0018 |

s.d.(AB) S(between runs): 0.0017 Sw(within run): 0.0013 S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B
0.118 - 0.142 for A-B

**REFERENCE MATERIAL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| KCN: | 35 | 0.10 | 0.0898 | -0.0102 | 0.0037 |
| FeCN: | 35 | 0.10 | 0.0891 | -0.0109 | 0.0062 |
| CLP Soil:(0.2) | 27 | 44.70 | 45.2706 | 0.5706 | 2.2436 |

**DUPLICATES:**

Aqueous Samples:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation (%) |
|---|---|---|---|
| 47 | 0 - 0.020 | 0.00035 | 27.4 |
| 2 | 0.021 - 0.040 | 0.00060 | 1.6 |
| 8 | 0.041 - 0.100 | 0.00048 | 0.6 |
| 11 | 0.101 - 0.200 | 0.00117 | 0.9 |
| 68 | Overall | 0.00059 | |

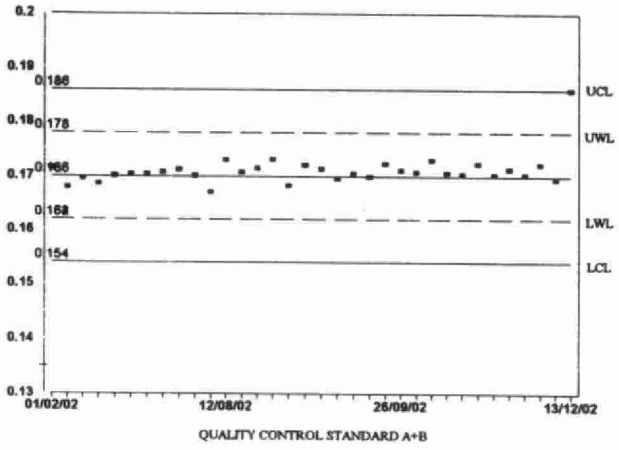
Soil Samples:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation (%) |
|---|---|---|---|
| 35 | 0 - 0.020 | 0.00014 | 5.3 |
| 2 | 0.021 - 0.040 | 0.00022 | 0.8 |
| 0 | 0.041 - 0.100 | N.A. | N.A. |
| 13 | 0.101 - 0.200 | 0.00159 | 0.1 |
| 50 | Overall | 0.00082 | |

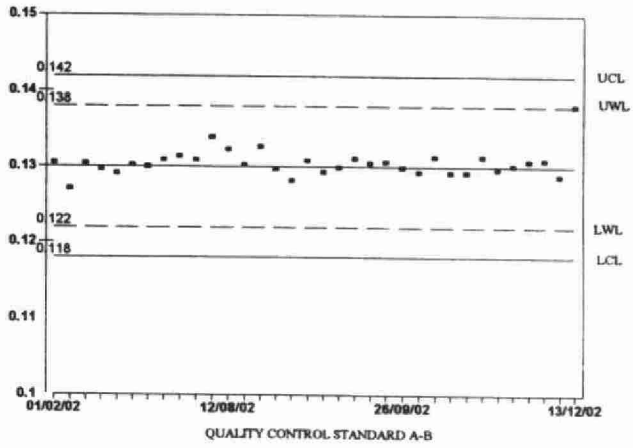
## **CYANIDE, TOTAL** (E3015)

QUALITY CONTROL DATA FROM 01/02/02 TO 12/13/02

Analytical Range: to 0.2 mg/L as CN




QUALITY CONTROL STANDARD A+B




QUALITY CONTROL STANDARD A-B

## FLUORIDE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | October 2001 |
| Method Reference No | E3172 | Reporting Unit | mg/L as F |
| LIMS Product Code | F3172, ANION3172, TCLPF3172 | Supervisor | P. WILSON |
| Sample Type/Matrix | Effluent, Industrial Waste, Process Water, Drinking Water, Ground Water, Leachate, Surface Water, Raw Sewage, Sediment, Dried Sludge, Unknown Material, Soil | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Plastic |

**ANALYTICAL PROCEDURE:**

Fluoride is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.0010 M sodium bicarbonate and 0.0035 M sodium carbonate with conductivity detector. The concentration of fluoride in mg/L as $F^-$ is determined by comparison of the sample scan to a series of standard scans.

**INSTRUMENTATION:**

Basic modular continuous flow ion chromatographic system, Justice Innovation ChromPerfect Spirit Data Station, plus control module (in-house design) for the automated sample introduction , timing and detector range switching.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |

**CALIBRATION:**

BL plus 9 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTB plus 3 standards, e.g. QCA |
| Drift | CHK1 and CHK2 standard approximately every 20 samples |

**NOTES:**

This method replaced E3369, October 2001.
Method E3369 calibration control values were used to establish initial control limits for the present method.
LIMS product code TCLPF3172 was added, October 2001.

## FLUORIDE (E3172)

QUALITY CONTROL DATA FROM 01/07/02 TO 12/27/02

Analytical Range: to 2.0 mg/L as F

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 87 | 1.60 | 1.61 | 0.01 | 0.0127 |
| B: | 87 | 0.80 | 0.81 | 0.01 | 0.0094 |
| C: | 87 | 0.16 | 0.16 | 0.00 | 0.0068 |
| A+B: | | 2.40 | 2.42 | 0.02 | 0.0186 |
| A-B: | | 0.80 | 0.80 | 0.00 | 0.0124 |
| B+C: | | 0.96 | 0.97 | 0.01 | 0.0123 |
| B-C: | | 0.64 | 0.65 | 0.01 | 0.0109 |

| | | | | |
|---|---|---|---|---|
| s.d.(AB) | S(between runs):0.0112 | Sw(within run): | 0.0087 | S/Sw: 1.3 |
| s.d.(BC) | S(between runs):0.0082 | Sw(within run): | 0.0077 | S/Sw: 1.1 |

The calibration is accepted if the calibration control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 2.262 | - | 2.538 | for | A+B |
| 0.697 | - | 0.903 | for | A-B |
| 0.877 | - | 1.043 | for | B+C |
| 0.578 | - | 0.702 | for | B-C |

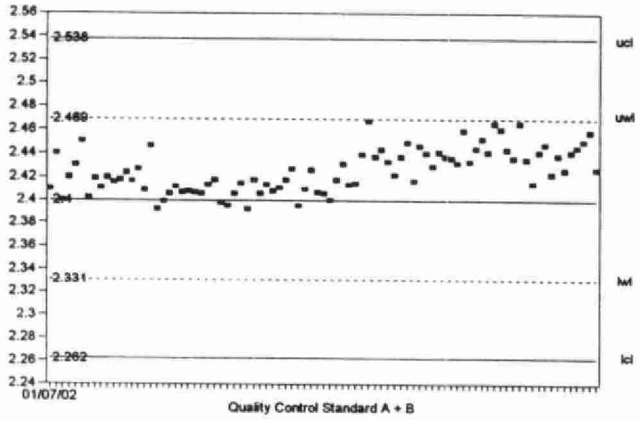
**DUPLICATES:**

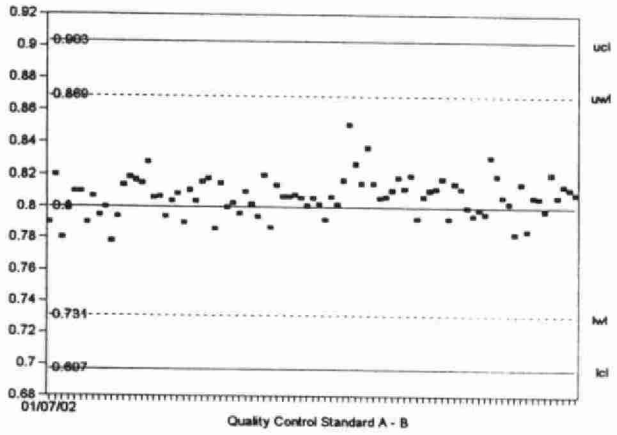
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 158 | 0.00 - 0.20 | 0.0064 | 8.7 |
| 23 | 0.21 - 0.40 | 0.0053 | 1.9 |
| 39 | 0.41 - 1.00 | 0.0081 | 1.3 |
| 12 | 1.01 - 2.00 | 0.0150 | 1.0 |
| 232 | Overall | 0.0073 | |

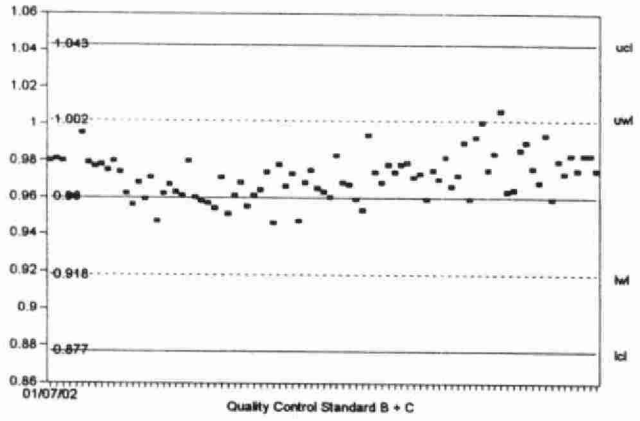
## FLUORIDE (E3172)

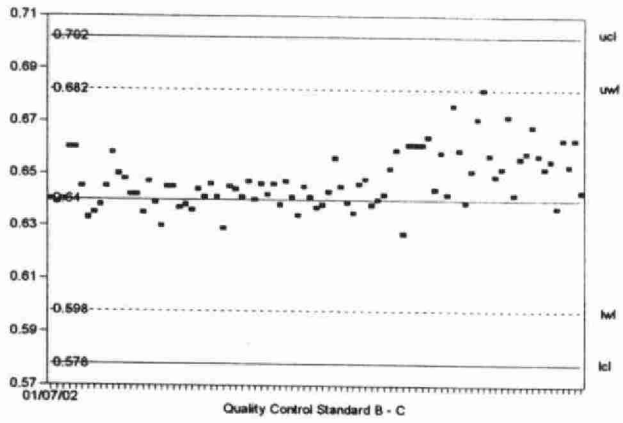
QUALITY CONTROL DATA FROM 01/07/02 TO 12/27/02

Analytical Range: to 2.0 mg/L as F



# NITRATE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3004 | Units | $\mu g/m^3$ as $NO_3$ |
| LIMS Product Code | ANION3004 | Supervisor | P. Wilson |
| Sample Type/Matrix | Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 3/4" or 1.9cm strip from 8"x10" filter |
| Container | 50 mL polypropylene tube |

## SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

## ANALYTICAL PROCEDURE:

Nitrate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of nitrate ( mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu g/m^3$ as $NO_3$.

Chloride and sulphate are determined simultaneously.

## INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.1 $\mu g/m^3$ | Current T value: 0.5 $\mu g/m^3$ |

**CALIBRATION:**
6 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | MB, IS(n), CS1, and CS2 |
| Drift | Duplicate plus 2 standards approximately every 20 samples |
| Recovery | CS3 & CS4 |

**NOTES:**

To convert unit from mg/L to µg/m$^3$, the final concentration of $NO_3$ in mg/L is multiplied by the following formula:

Result (mg/L) X 50mL X (63/6.75) / air volume = µg/m$^3$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

**NITRATE** (E3004)

QUALITY CONTROL DATA FOR 01/17/02 TO 12/31/02

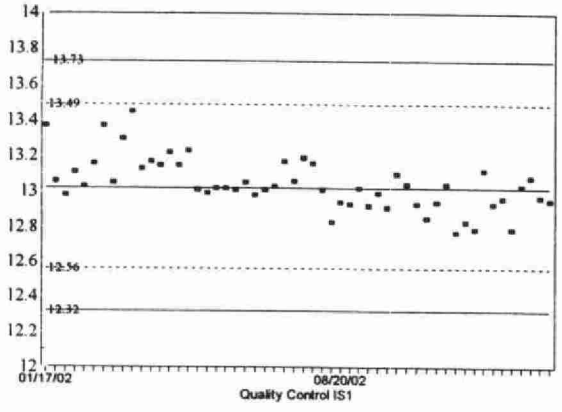
Analytical Range: to 28.61 $\mu g/m^3$ as $NO_3$

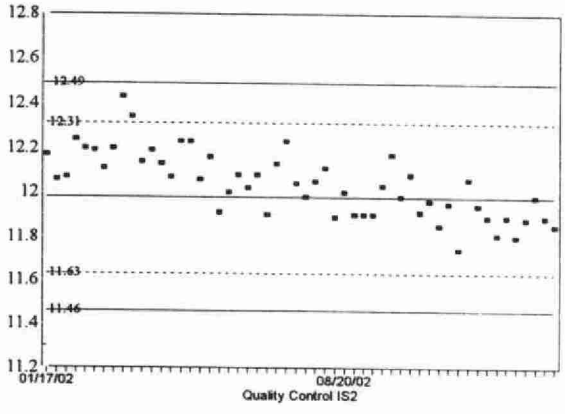
**DUPLICATES:**

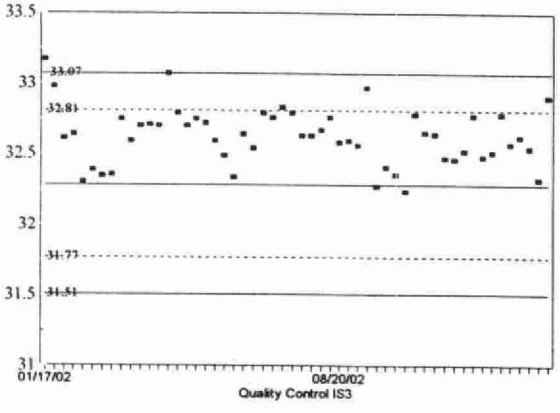
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 64 | 0.00 - 2.86 | 0.0552 | 7.4 |
| 9 | 2.89 - 7.15 | 0.0782 | 2.4 |
| 5 | 7.18 - 14.31 | 0.2074 | 2.2 |
| 0 | 14.33 - 28.61 | N.A. | N.A. |
| 78 | Overall | 0.0772 | |

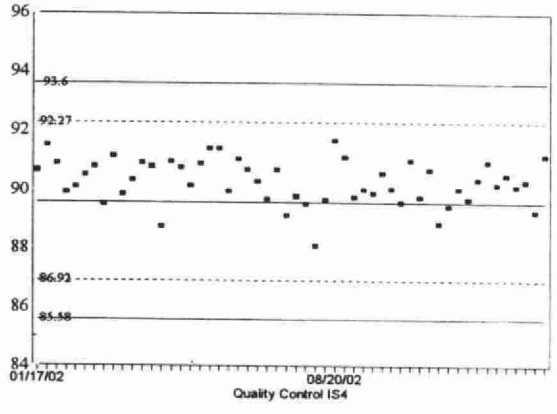
## NITRATE (E3004)

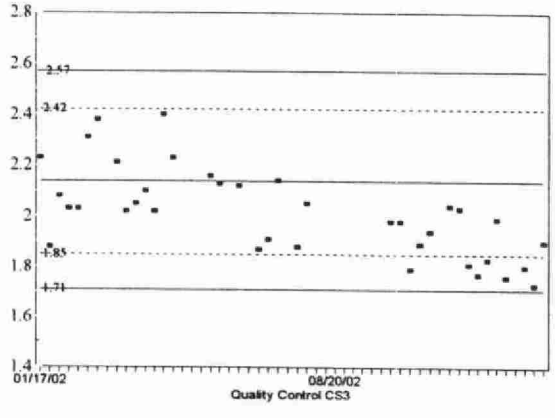
QUALITY CONTROL DATA FROM 01/17/02 TO 12/31/02
Analytical Range For IS Controls: to 100 mg/L
Analytical Range For CS Controls: to 28.61 µg/m$^3$

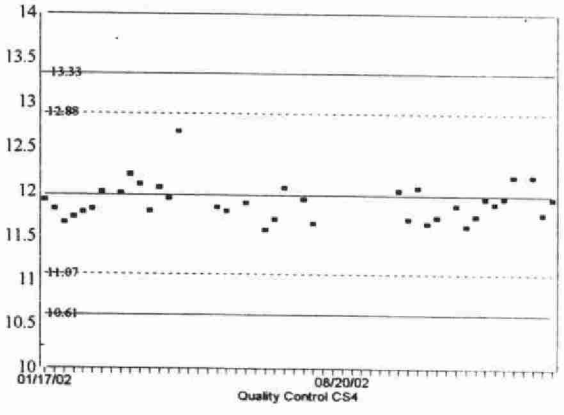



Note: For explanation of any exceedence, refer to raw data file.

# NITRILOTRIACETIC ACID

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 35901 |
| Method Reference No. | E3406 | Reporting Unit | mg/L as NTA |
| LIMS Product Code | NTA3406, TCLPNTA3406 | Supervisor | P. Wilson |
| Sample Type/Matrix | Drinking Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrilotriacetic Acid is separated from other anions in the samples by automated suppressed gradient ion chromatography. A sodium hydroxide eluent is used with conductivity detection. The concentration of Nitrilotriacetic acid in mg/L as NTA is determined by comparison of the sample scan to a series of standard scans.

## INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system with gradient flow control module .

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.01 | Current T value: 0.05 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 2 standards, e.g. QCA |
| Drift | 1 standard every 10 samples |

## NOTE:

LIMS product code TCLPNTA3406 was added, April 2001.

## NITRILOTRIACETIC ACID (E3406)

QUALITY CONTROL DATA FROM 01/17/02 TO 12/19/02

Analytical Range: to 1.00 mg/L as NTA

### CALIBRATION CONTROL:

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 23 | 0.80 | 0.799 | -0.001 | 0.0157 |
| B: | 23 | 0.20 | 0.201 | 0.001 | 0.0142 |
| A+B: | | 1.00 | 1.000 | 0.003 | 0.0257 |
| A-B: | | 0.60 | 0.597 | -0.003 | 0.0154 |

s.d.(AB) S(between runs): 0.0153 Sw(within run): 0.0111 S/Sw: 1.38

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.95 - 1.05 for A+B
0.56 - 0.64 for A-B

### DUPLICATES:

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 14 | 0.00 - 0.10 | 0.0080 | 9.6 |
| 38 | 0.10 - 0.20 | 0.0127 | 11.4 |
| 0 | 0.20 - 0.50 | N.A. | N.A. |
| 0 | 0.50 - 1.00 | N.A. | N.A. |
| 52 | Overall | | |

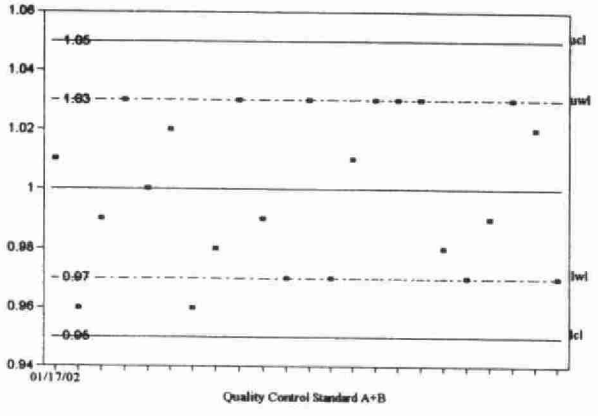
### OTHER CHECKS:

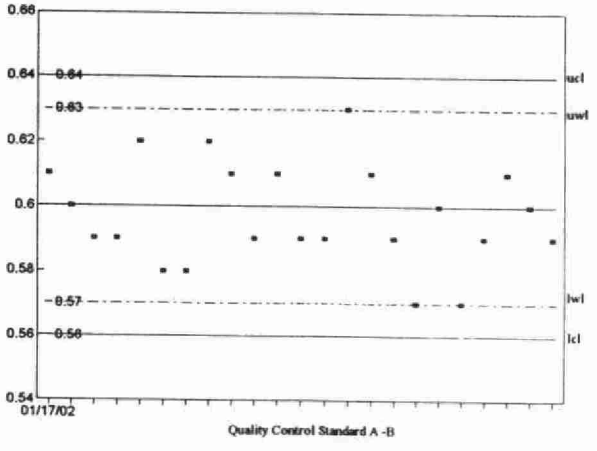
| | n | Mean | Standard Deviation (1) |
|---|---|---|---|
| Long Term Blank | 23 | 0 | 0 |

## NITRILOTRIACETIC ACID (E3406)

QUALITY CONTROL DATA FROM 01/17/02 TO 12/19/02

Analytical Range: to 1.00 mg/L as NTA



## NITROGEN, AMMONIA PLUS AMMONIUM

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3364 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3364 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.
Approximate absorbance: 0.5 at the full scale level.
Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system plus the following modules: 2 of 38$^0$C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.
Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.002 | Current T value: 0.010 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL, standard , and BL after every 10 samples |

**NOTES:**

The HP data capture / processing system was replaced by Labtronics in August 1999.

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 89 | 1.6 | 1.598 | -0.002 | 0.010 |
| **B** | 89 | 0.8 | 0.795 | -0.005 | 0.007 |
| **C** | 89 | 0.16 | 0.158 | -0.002 | 0.004 |
| **A + B** | | 2.4 | 2.393 | -0.007 | 0.013 |
| **A - B** | | 0.8 | 0.803 | 0.003 | 0.012 |
| **B + C** | | 0.96 | 0.952 | -0.008 | 0.009 |
| **B - C** | | 0.64 | 0.637 | -0.003 | 0.007 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.009 |
| | Within Runs | 0.0085 |
| | Between/Within | 1.0588 |
| s.d.(BC) | Between Runs | 0.0058 |
| | Within Runs | 0.0049 |
| | Between/Within | 1.1837 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 2.424 | 2.376 | 2.447 | 2.353 |
| A - B | 0.824 | 0.776 | 0.835 | 0.765 |
| B + C | 0.974 | 0.946 | 0.989 | 0.931 |
| B - C | 0.654 | 0.626 | 0.662 | 0.618 |

**DUPLICATES**

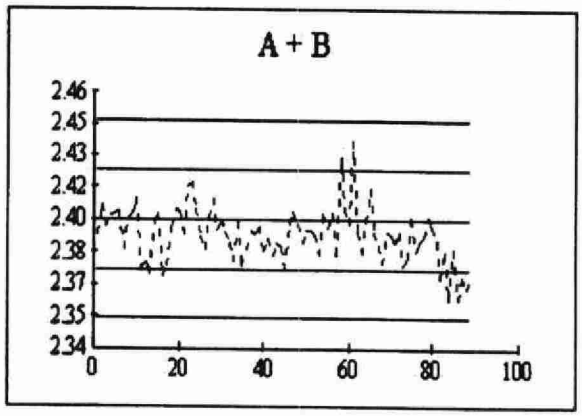
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 225 | 0 - 10% | 0.004 | 17.4 |
| 8 | 10 - 20% | 0.007 | 2.6 |
| 6 | 20 - 50% | 0.016 | 2.9 |
| 1 | 50 - 100% | 0.009 | 0.6 |
| 240 | Total | 0.005 | 9.9 |

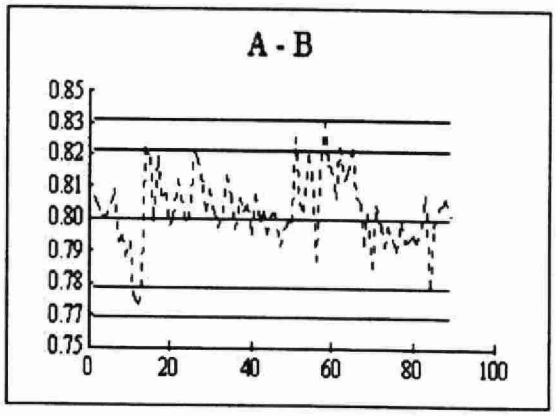
**OTHER CHECKS**

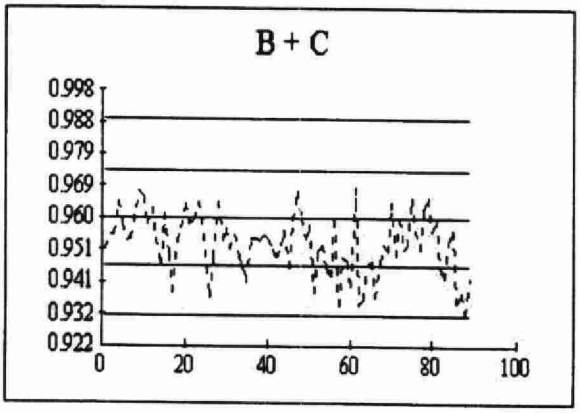
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 89 | 0.002 | 0.005 |

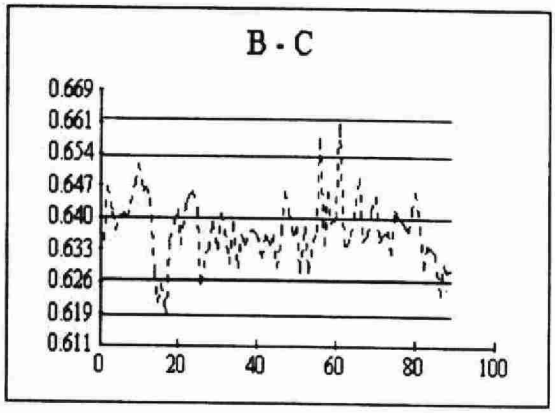
Nitrogen; ammonia+ammonium [ E3364A ]

QC Data; 1/1/02 to 12/31/02



## NITROGEN, AMMONIA PLUS AMMONIUM

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/77 |
| Method Reference No. | E3366 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3366 | Supervisor | P.Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Ground Water. | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.
Approximate absorbance: 0.7 at the full scale level.
Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system plus one 38$^0$C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL , standard and BL every 10 samples |

**NOTES:**

The HP capture / processing system was replaced by Labtronics in October 1999.

**Nitrogen; ammonia+ammonium (E3366)**

**Analytical Range: to 50.0 mg/L as N**

**CALIBRATION CONTROL**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 54 | 40 | 39.96 | -0.04 | 0.188 |
| **B** | 54 | 20 | 20.091 | 0.091 | 0.118 |
| **C** | 54 | 4 | 3.988 | -0.012 | 0.089 |
| **A + B** | | 60 | 60.051 | 0.051 | 0.243 |
| **A - B** | | 20 | 19.869 | -0.131 | 0.198 |
| **B + C** | | 24 | 24.079 | 0.079 | 0.156 |
| **B - C** | | 16 | 16.103 | 0.103 | 0.138 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.1566 |
| | Within Runs | 0.14 |
| | Between/Within | 1.1186 |
| s.d.(BC) | Between Runs | 0.1041 |
| | Within Runs | 0.0976 |
| | Between/Within | 1.0666 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 60.62 | 59.38 | 61.2 | 58.76 |
| A - B | 20.62 | 19.38 | 20.9 | 19.07 |
| B + C | 24.33 | 23.67 | 24.7 | 23.34 |
| B - C | 16.33 | 15.67 | 16.5 | 15.5 |

**DUPLICATES**

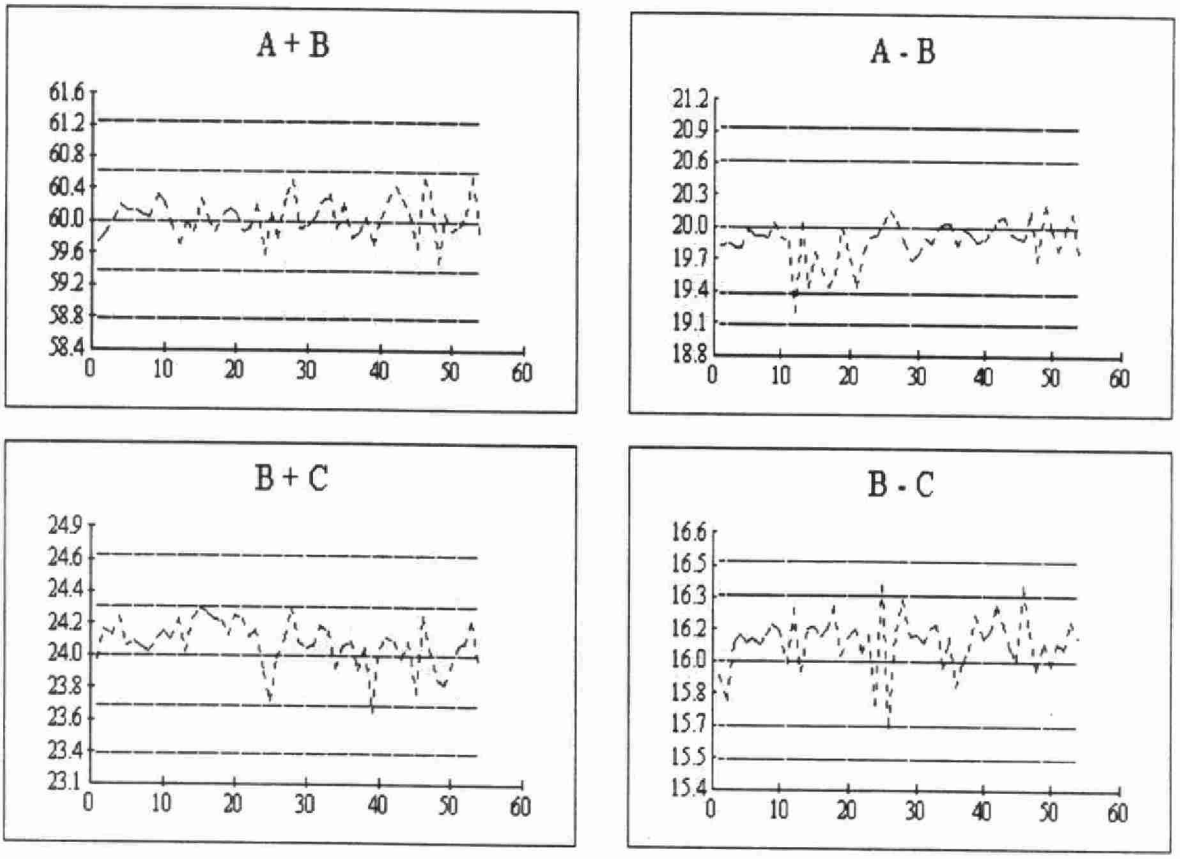
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 95 | 0 - 10% | 0.041 | 7.5 |
| 16 | 10 - 20% | 0.056 | 0.7 |
| 15 | 20 - 50% | 0.092 | 0.6 |
| 7 | 50 - 100% | 0.714 | 2 |
| 133 | Total | 0.171 | |

**OTHER CHECKS**

| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 54 | -0.023 | 0.031 |

Nitrogen; ammonia+ammonium ( E3366A )

QC Data; 1/1/02 to 12/31/02



# NITROGEN, NITRATE PLUS NITRITE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3364 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3364 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL , standard and BL every 10 samples |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step. |

**NOTES:** The HP data capture / processing system was replaced by Labtronics in August 1999.

## Nitrogen; nitrate+nitrite (E3364)

### Analytical Range: to 5.00 mg/L as N

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| A | 91 | 4 | 3.994 | -0.006 | 0.032 |
| B | 91 | 2 | 2.003 | 0.003 | 0.019 |
| C | 91 | 0.4 | 0.396 | -0.004 | 0.007 |
| A + B | | 6 | 5.998 | -0.002 | 0.041 |
| A - B | | 2 | 1.991 | -0.009 | 0.034 |
| B + C | | 2.4 | 2.3996 | -0.0004 | 0.022 |
| B - C | | 1.6 | 1.607 | .007 | 0.019 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0264 |
| | Within Runs | 0.024 |
| | Between/Within | 1.1 |
| s.d.(BC) | Between Runs | 0.0146 |
| | Within Runs | 0.0134 |
| | Between/Within | 1.0896 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 6.066 | 5.934 | 6.132 | 5.868 |
| A - B | 2.066 | 1.934 | 2.099 | 1.901 |
| B + C | 2.436 | 2.364 | 2.472 | 2.328 |
| B - C | 1.636 | 1.564 | 1.654 | 1.546 |

**DUPLICATES**

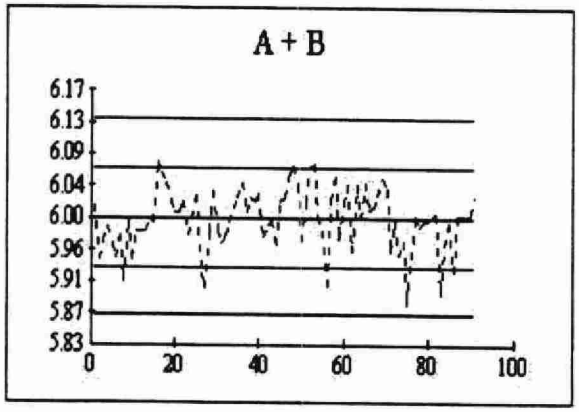
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 161 | 0 - 10% | 0.006 | 3.1 |
| 28 | 10 - 20% | 0.012 | 1.7 |
| 34 | 20 - 50% | 0.017 | 1 |
| 24 | 50 - 100% | 0.033 | 1 |
| 247 | Total | 0.013 | |

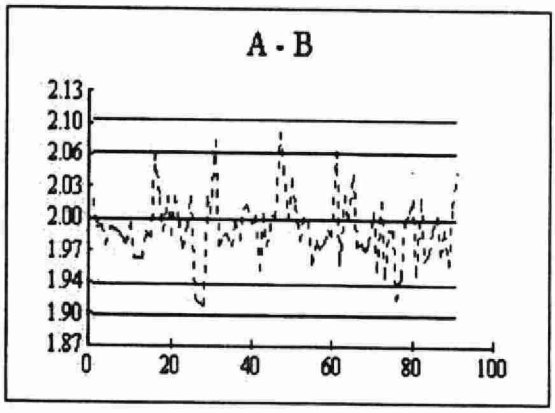
**OTHER CHECKS**

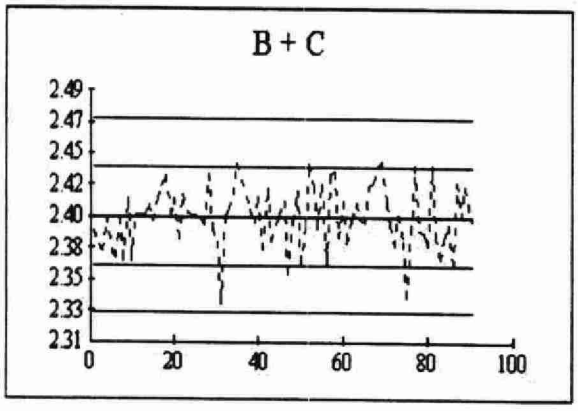
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 91 | -0.001 | 0.004 |

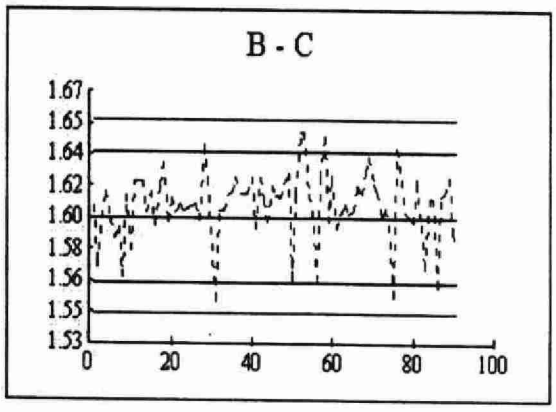
Nitrogen; nitrate+nitrite ( E3364A )

QC Data; 1/1/02 to 12/31/02



# NITROGEN, NITRATE PLUS NITRITE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3366 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3366,TCLPNOT3366 | Supervisor | P.Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Ground Water. | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.7 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive phosphate are determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter. Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL ,standard and BL every 10 samples |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50mg/L) confirms effective interference suppression. |
| Recovery | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step. |

**NOTES:** The HP capture / processing system was replaced by Labtronics in October 1999.
LIMS product code TCLPNOT3366 was added in April 2001.

**Nitrogen; nitrate+nitrite (E3366)**

**Analytical Range: to 50.0 mg/L as N**

**CALIBRATION CONTROL**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 54 | 40 | 39.905 | -0.095 | 0.215 |
| **B** | 54 | 20 | 20.093 | 0.093 | 0.127 |
| **C** | 54 | 4 | 3.979 | -0.021 | 0.071 |
| **A + B** | | 60 | 59.998 | -0.002 | 0.262 |
| **A - B** | | 20 | 19.812 | -0.188 | 0.235 |
| **B + C** | | 24 | 24.072 | 0.072 | 0.17 |
| **B - C** | | 16 | 16.115 | 0.115 | 0.116 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.1762 |
| | Within Runs | 0.1662 |
| | Between/Within | 1.0602 |
| s.d.(BC) | Between Runs | 0.1027 |
| | Within Runs | 0.082 |
| | Between/Within | 1.2524 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 60.67 | 59.33 | 61.3 | 58.66 |
| A - B | 20.67 | 19.33 | 21 | 18.99 |
| B + C | 24.36 | 23.64 | 24.7 | 23.28 |
| B - C | 16.36 | 15.64 | 16.5 | 15.46 |

**DUPLICATES**

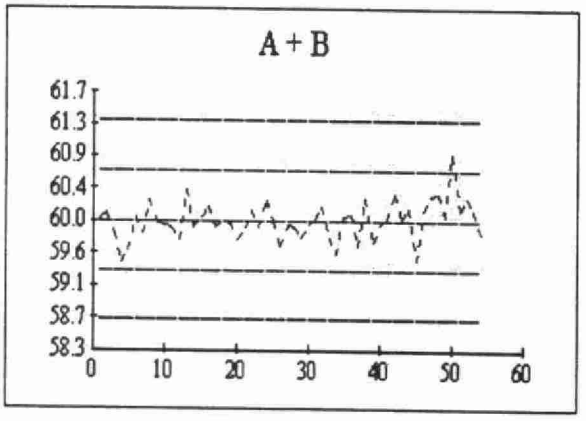
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 106 | 0 - 10% | 0.041 | 4.6 |
| 12 | 10 - 20% | 0.07 | 1 |
| 21 | 20 - 50% | 0.159 | 1.1 |
| 9 | 50 - 100% | 0.232 | 0.7 |
| 148 | Total | 0.092 | 1.7 |

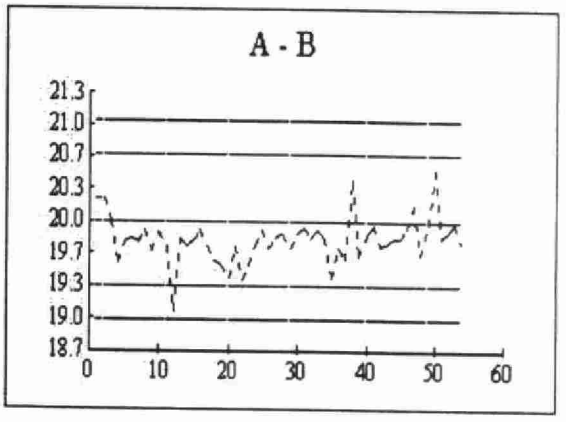
**OTHER CHECKS**

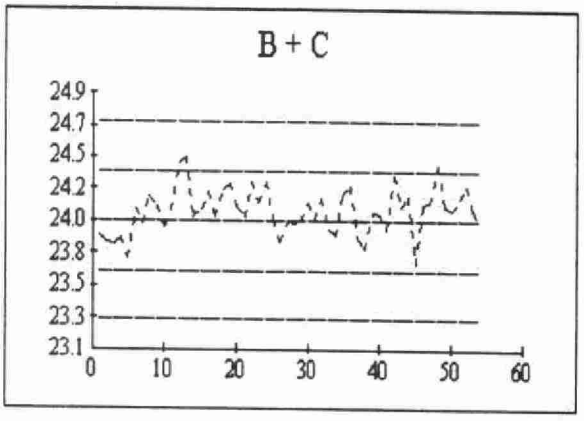
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 54 | -0.025 | 0.023 |

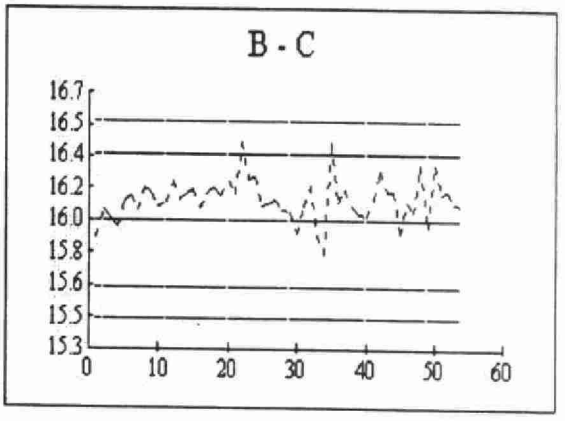
Nitrogen; nitrate+nitrite ( E3366A )

QC Data; 1/1/02 to 12/31/02



# NITROGEN, NITRITE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3364 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3364 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride.
Approximate absorbance: 0.6 at the full scale level.
Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.
Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.001 | Current T value: 0.005 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL , standard and BL after every 10 samples |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step. |

## NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

## Nitrogen; nitrite (E3364)

### Analytical Range: to 0.200 mg/L as N

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 87 | 0.16 | 0.1593 | -0.0007 | 0.0012 |
| **B** | 87 | 0.08 | 0.0801 | 0.0001 | 0.0011 |
| **C** | 87 | 0.016 | 0.0162 | 0.0002 | 0.0007 |
| **A + B** | | 0.24 | 0.2394 | -0.0006 | 0.0018 |
| **A - B** | | 0.08 | 0.0792 | -0.0008 | 0.0014 |
| **B + C** | | 0.096 | 0.0963 | 0.0003 | 0.0014 |
| **B - C** | | 0.064 | 0.0639 | -0.0001 | 0.0012 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0011 |
| | Within Runs | 0.001 |
| | Between/Within | 1.1 |
| s.d.(BC) | Between Runs | 0.0009 |
| | Within Runs | 0.0008 |
| | Between/Within | 1.1 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 0.243 | 0.237 | 0.245 | 0.235 |
| A - B | 0.083 | 0.077 | 0.084 | 0.076 |
| B + C | 0.098 | 0.094 | 0.101 | 0.091 |
| B - C | 0.066 | 0.062 | 0.067 | 0.061 |

**DUPLICATES**

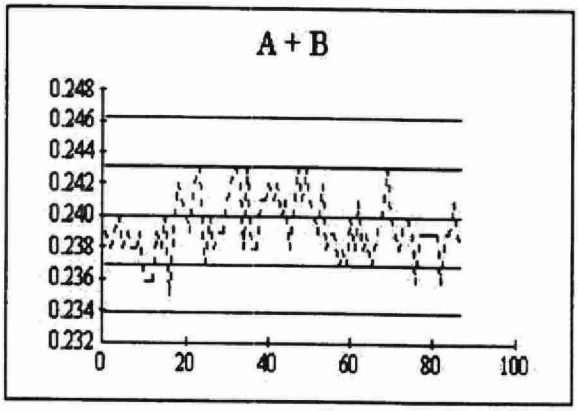
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 178 | 0 - 10% | 0.001 | 19.4 |
| 12 | 10 - 20% | 0.001 | 3.3 |
| 13 | 20 - 50% | 0.001 | 1.6 |
| 4 | 50 - 100% | 0.004 | 2.9 |
| 206 | Total | 0.001 | |

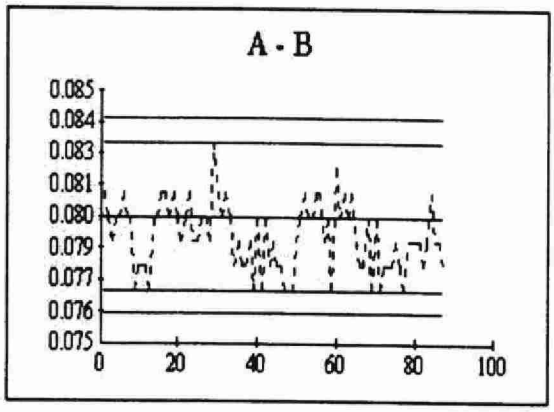
**OTHER CHECKS**

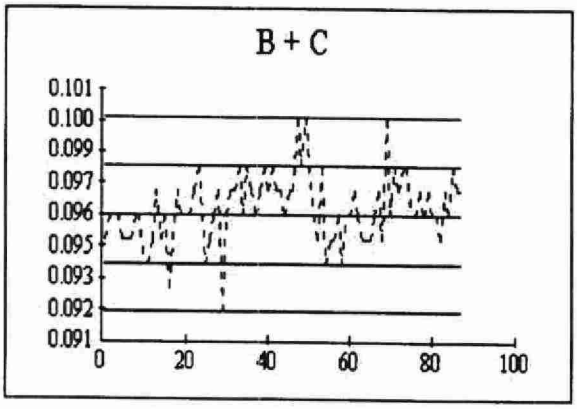
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 87 | 0.0004 | 0.001 |

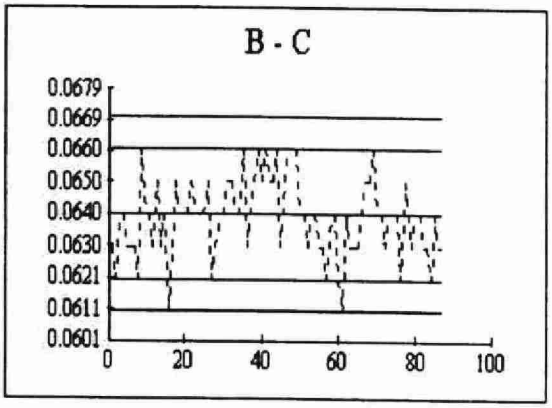
Nitrogen; nitrite [ E3364A ]

QC Data; 1/1/02 to 12/31/02



# NITROGEN, NITRITE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No | E3366 | Reporting Unit | mg/L as N |
| LIMS Product Code | DISNUT3366 | Supervisor | P.Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Ground Water. | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride.
Approximate absorbance: 0.3 at the full scale level.
Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm. Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.005 | Current T value: 0.025 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL ,standard and BL every 10 samples |
| Interference | Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression. |
| Recovery | Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step. |

## NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

**Nitrogen; nitrite (E3366)**

**Analytical Range: to 2.00 mg/L as N**

**CALIBRATION CONTROL**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 54 | 1.6 | 1.602 | 0.002 | 0.011 |
| **B** | 54 | 0.8 | 0.804 | 0.004 | 0.006 |
| **C** | 54 | 0.16 | 0.16 | 0.000 | 0.006 |
| **A + B** | | 2.4 | 2.406 | 0.006 | 0.015 |
| **A - B** | | 0.8 | 0.798 | 0.002 | 0.009 |
| **B + C** | | 0.96 | 0.964 | 0.004 | 0.010 |
| **B - C** | | 0.64 | 0.644 | 0.004 | 0.006 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0085 |
| | Within Runs | 0.0064 |
| | Between/Within | 1.3281 |
| s.d.(BC) | Between Runs | 0.0058 |
| | Within Runs | 0.0042 |
| | Between/Within | 1.381 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 2.424 | 2.376 | 2.448 | 2.353 |
| A - B | 0.824 | 0.776 | 0.836 | 0.764 |
| B + C | 0.972 | 0.948 | 0.984 | 0.936 |
| B - C | 0.652 | 0.628 | 0.658 | 0.622 |

**DUPLICATES**

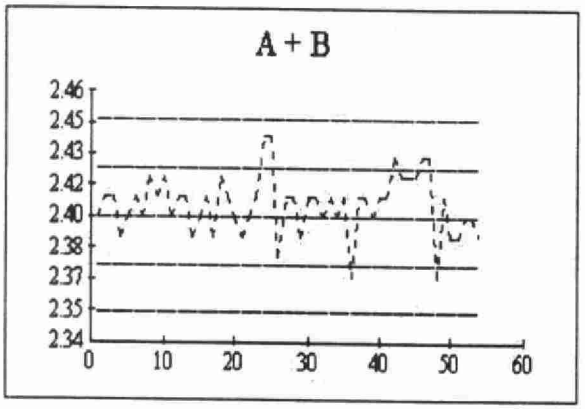
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 72 | 0 - 10% | 0.005 | 8.4 |
| 11 | 10 - 20% | 0.012 | 4 |
| 12 | 20 - 50% | 0.018 | 2.5 |
| 6 | 50 - 100% | 0.023 | 1.5 |
| 101 | Total | 0.01 | 3.9 |

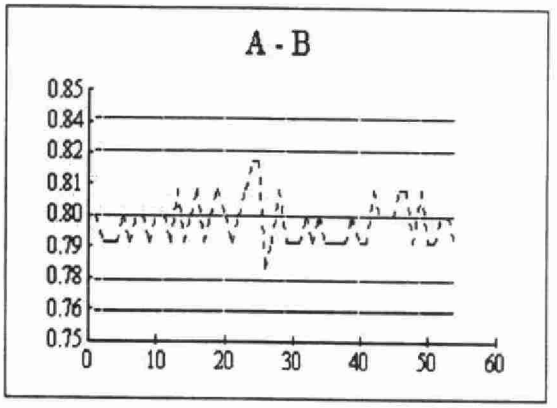
**OTHER CHECKS**

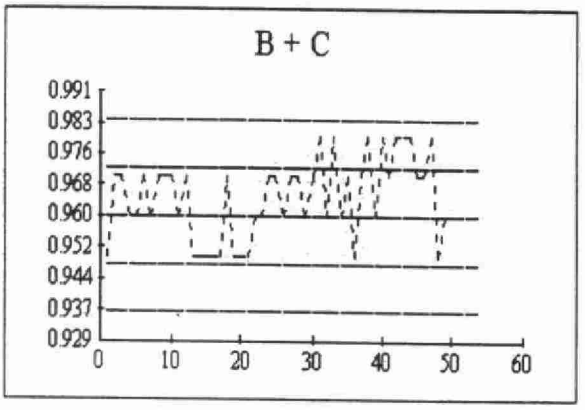
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 54 | 0.000 | 0.002 |

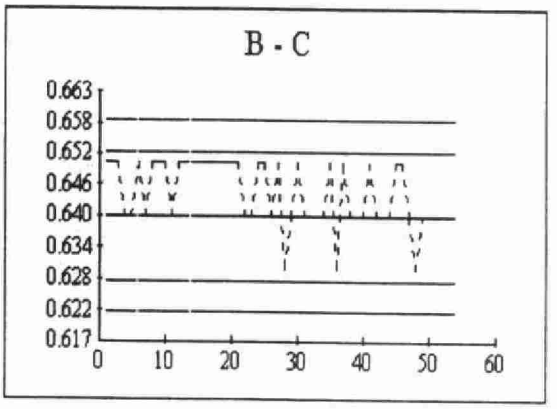
**Nitrogen; nitrite ( E3366A )**

**QC Data; 1/1/02 to 12/31/02**



# NITROGEN, TOTAL KJELDAHL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Mar '89 |
| Method Reference No. | E3116 | Reporting Unit | mg/g as N |
| LIMS Product Code | TNP3116 | Supervisor | P. Wilson |
| Sample Type/Matrix | Soil, Sediment, Dried Sludge | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 0.08 to 0.4 g |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

## INSTRUMENTATION:

Hot plate .

Basic automated modular continuous flow system : 37.5$^0$C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.1 | Current T value: 0.5 |

## CALIBRATION:

3 High and 2 Low Calibration Standards

## CONTROLS:

| | |
|---|---|
| Calibration | In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92) |
| Drift | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery | 1 digested BL plus 4 digested standards |

## NOTES:

System is calibrated with undigested standards.

Low Recoveries for the Domestic Sludge SRM 2781 are under investigation.

## NITROGEN, TOTAL KJELDAHL (E3116)

QUALITY CONTROL DATA FROM 01/29/02 TO 12/04/02

Analytical Range: to 10 mg/L as N

**QUALITY CONTROL:**

| | n | Expected Concentration (mg/g) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| RS92 - In House Soil Composite | 20 | 1.69 | 1.57 | 0.1047 |
| RSM-2781 -Domestic Sludge Certified | 20 | 42.2 | 44.85 | 1.3606 |

The run is accepted if the control values obtained lie within the ranges:

1.39 - 1.99 for RS92

29.1 - 55.2 for RSM-2781

**Recovery Standards**

| | n | Expected Concentration (mg/L) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| R1 | 20 | 5.25 | 5.29 | 0.1559 |
| R2 | 20 | 1.75 | 1.71 | 0.0885 |

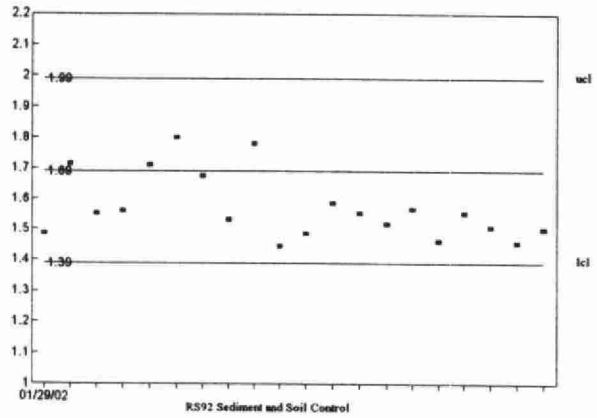
**DUPLICATES**: (Sediment/Soils)

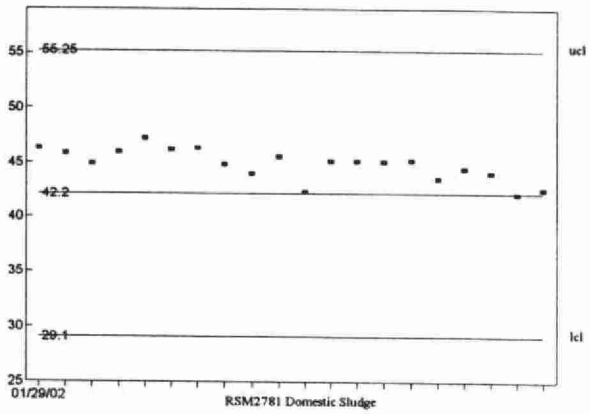
| n Data Pairs | Sample Concentration Span (mg/g) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 32 | 0.00 - 2.00 | 0.1026 | 8.5 |
| 15 | 2.01 - 4.00 | 0.2019 | 7.6 |
| 7 | 4.01 - 10.0 | 0.0815 | 1.6 |
| 1 | 10.1 - 20.0 | N.A. | N.A. |
| 55 | Overall | 0.1545 | |

## NITROGEN, TOTAL KJELDAHL (E3116)

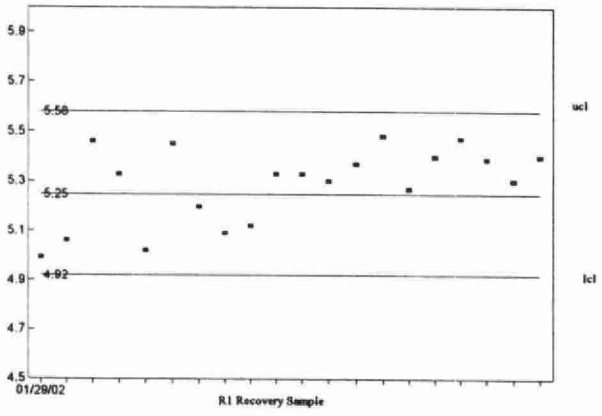
QUALITY CONTROL DATA FROM 01/29/02 TO 12/04/02

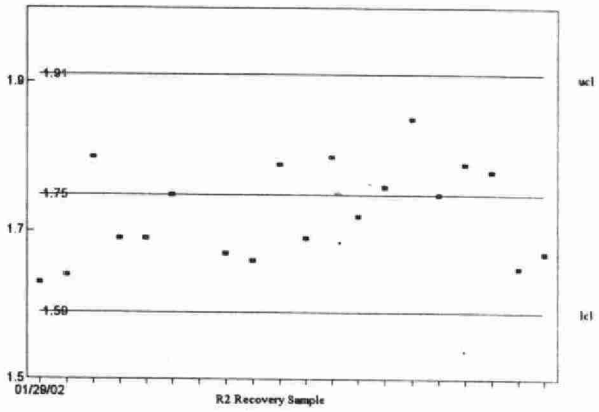
Analytical Range: to 10 mg/g as N




Analytical Range: to 10 mg/L as N



# NITROGEN, TOTAL KJELDAHL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Mar '89 |
| Method Reference No. | E3118 | Reporting Unit | mg/g as N |
| LIMS Product Code | TNP3118 | Supervisor | P. Wilson |
| Sample Type/Matrix | Vegetation, Moss Bag | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 0.02 to 0.04 g |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

## INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : 37.5$^{0}$C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.20 | Current T value: 1.00 |

## CALIBRATION:

3 High and 2 Low Calibration Standards

## CONTROLS:

| | |
|---|---|
| Calibration | In house composite A-VEG, plus QC VEG (Pine Needles) |
| Drift | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery | 1 digested BL plus 4 digested standards |

## NOTES:

System is calibrated with undigested standards.

## NITROGEN, TOTAL KJELDAHL (E3118)

QUALITY CONTROL DATA FROM 03/05/02 TO 12/04/02

Analytical Range: to 10 mg/L as N

**QUALITY CONTROL:**

| | n | Expected Concentration (mg/g) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| Pine Needles | 7 | 12.1 | 11.46 | 0.4725 |

The run is accepted if the control values obtained lie within the ranges:
10.3 - 13.9 for Pine Needles

**Recovery Standards**

| | n | Expected Concentration (mg/L) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| R1 | 7 | 5.25 | 5.31 | 0.1626 |
| R2 | 7 | 1.75 | 1.70 | 0.0608 |

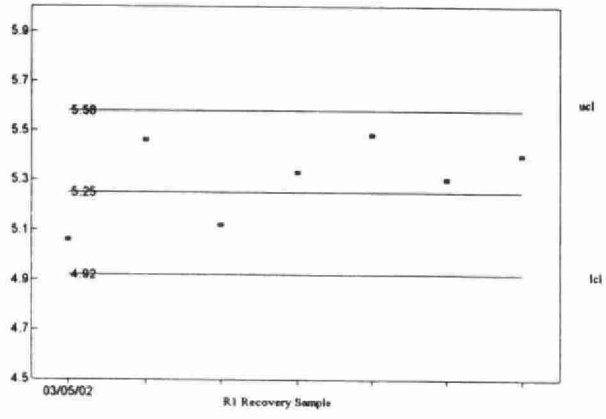
**DUPLICATES**: (VEGETATION)

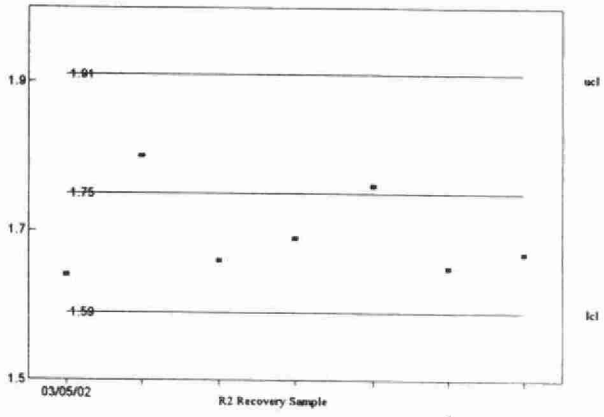
| n Data Pairs | Sample Concentration Span (mg/g) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 3 | 0.00 - 10.0 | 0.4166 | 4.3 |
| 5 | 10.1 - 20.0 | 0.5907 | 3.7 |
| 11 | 20.1 - 50.0 | 1.1992 | 4.2 |
| 19 | Overall | 0.9756 | |

## NITROGEN, TOTAL KJELDAHL (E3118)

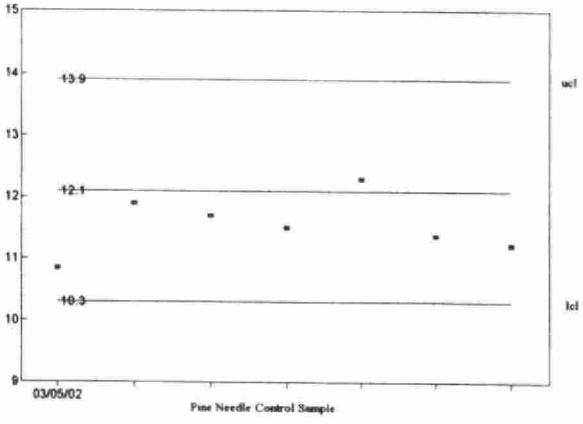
QUALITY CONTROL DATA FROM 04/05/02 TO 12/04/02

Analytical Range : to 10 mg/Las N




Analytical Range: to 10 mg/g as N



# NITROGEN, TOTAL KJELDAHL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No. | E3367 | Reporting Unit | mg/L as N |
| LIMS Product Code | TOTNUT3367 | Supervisor | P.Wilson |
| Sample Type/Matrix | Precipitation, Drinking Water, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.3 at the full scale level.

Total phosphorus is determined simultaneously.

## INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

Data capture and processing via a computer system

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

## CALIBRATION:

BL plus 7 undigested standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA |
| Drift | BL, undigested standard , BL every 10 samples |
| Recovery | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

## NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999.

## Nitrogen;total Kjeldahl (E3367)

### Analytical Range: to 2.00 mg/L as N

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 79 | 1.6 | 1.601 | 0.001 | 0.010 |
| **B** | 79 | 0.8 | 0.801 | 0.001 | 0.005 |
| **C** | 79 | 0.16 | 0.16 | 0.000 | 0.006 |
| **A + B** | | 2.4 | 2.402 | 0.002 | 0.012 |
| **A - B** | | 0.8 | 0.8 | 0.000 | 0.011 |
| **B + C** | | 0.96 | 0.961 | 0.001 | 0.008 |
| **B - C** | | 0.64 | 0.641 | 0.001 | 0.008 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0083 |
| | Within Runs | 0.0078 |
| | Between/Within | 1.0641 |
| s.d.(BC) | Between Runs | 0.0054 |
| | Within Runs | 0.0057 |
| | Between/Within | 0.9474 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 2.44 | 2.36 | 2.48 | 2.32 |
| A - B | 0.84 | 0.76 | 0.86 | 0.74 |
| B + C | 0.984 | 0.936 | 1.007 | 0.913 |
| B - C | 0.663 | 0.617 | 0.675 | 0.605 |

**DUPLICATES**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 73 | 0 - 10% | 0.010 | 8.1 |
| 89 | 10 - 20% | 0.012 | 4.1 |
| 65 | 20 - 50% | 0.018 | 3.1 |
| 6 | 50 - 100% | 0.024 | 1.8 |
| 233 | Total | 0.014 | |

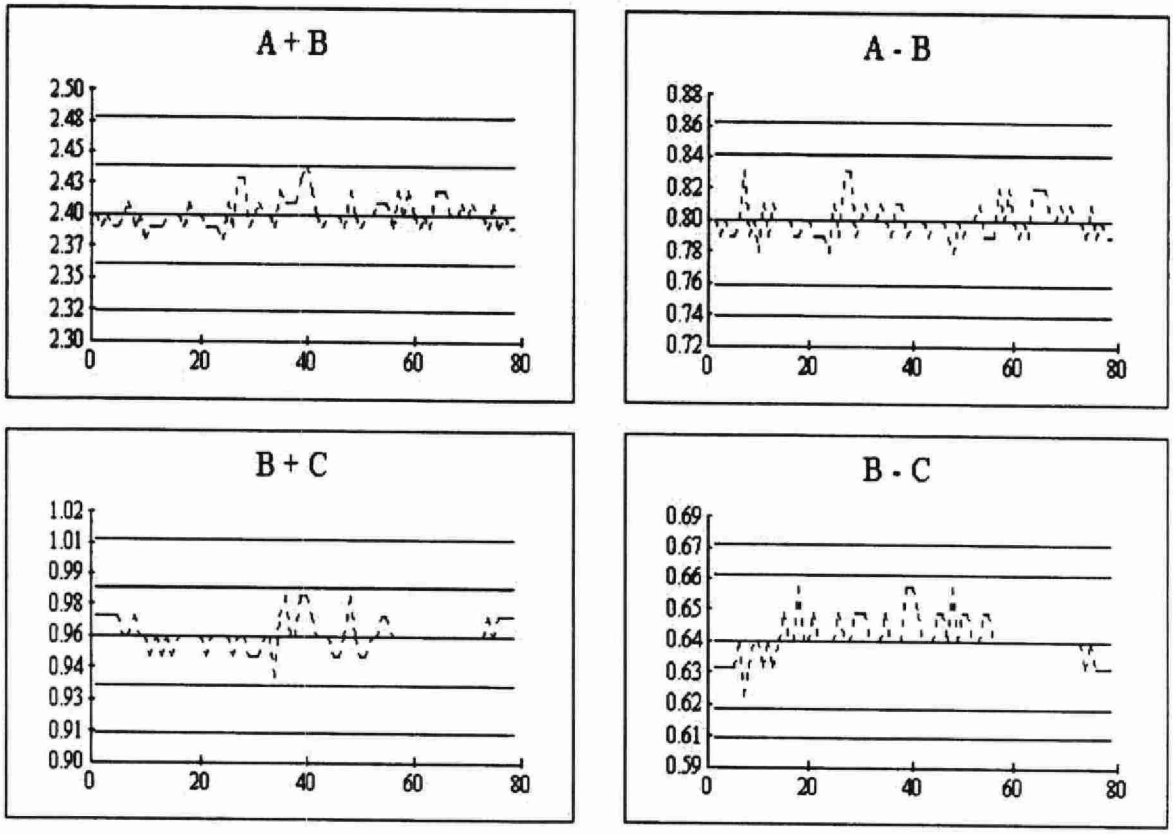
**RECOVERIES**

| *Number* | *Expected* | *Mean* | *Std. Dev.* |
|---|---|---|---|
| 79 | 1.4 | 1.399 | 0.024 |
| 79 | 0.84 | 0.841 | 0.012 |
| 79 | 0.28 | 0.282 | 0.022 |

| **OTHER CHECKS** | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 79 | 0.002 | 0.007 |
| Digested Blank | 79 | 0.012 | 0.009 |

**Nitrogen;total Kjeldahl ( E3367A )**

**QC Data: 01/01/02 to 12/31/02**



# NITROGEN, TOTAL KJELDAHL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No | E3368 | Reporting Unit | mg/L as N |
| LIMS Product Code | TOTNUT3368 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Raw Sewage, Industrial Waste, Effluent, Ground Water, Process Water, Leachate. | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.
Approximate absorbance: 1.1 at the full scale level.
Total phosphorus is determined simultaneously.

## INSTRUMENTATION:

Three block digesters
Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.
Data capture and processing via a computer system

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |

## CALIBRATION:

BL plus 7 undigested standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA |
| Drift | BL every 10 samples; undigested standard every 20 samples |
| Recovery | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

## NOTES:

System is calibrated with undigested standards.
The HP capture / processing system was replaced by Labtronics in April 1999.

**Nitrogen; total Kjeldahl (E3368)**

**Analytical Range: to 50.0 mg/L as N**

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 53 | 40 | 39.965 | -0.035 | 0.152 |
| **B** | 53 | 20 | 20.065 | 0.065 | 0.100 |
| **C** | 53 | 4 | 3.947 | -0.053 | 0.060 |
| **A + B** | | 60 | 60.03 | 0.030 | 0.230 |
| **A - B** | | 20 | 19.9 | -0.100 | 0.115 |
| **B + C** | | 24 | 24.012 | 0.012 | 0.122 |
| **B - C** | | 16 | 16.118 | 0.118 | 0.110 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.1287 |
| | Within Runs | 0.0813 |
| | Between/Within | 1.583 |
| s.d.(BC) | Between Runs | 0.0822 |
| | Within Runs | 0.0778 |
| | Between/Within | 1.0566 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 60.36 | 59.64 | 60.73 | 59.27 |
| A - B | 20.36 | 19.64 | 20.55 | 19.45 |
| B + C | 24.21 | 23.79 | 24.42 | 23.58 |
| B - C | 16.21 | 15.79 | 16.32 | 15.68 |

**DUPLICATES**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 129 | 0 - 10% | 0.082 | 8.8 |
| 12 | 10 - 20% | 0.257 | 3.8 |
| 11 | 20 - 50% | 0.286 | 1.9 |
| 1 | 50 - 100% | N.A. | N.A. |
| 153 | Total | 0.135 | |

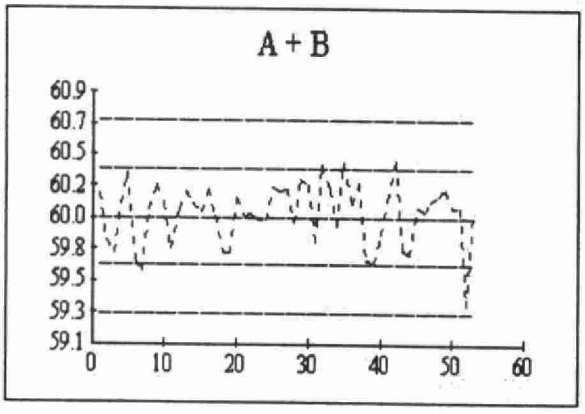
**RECOVERIES**

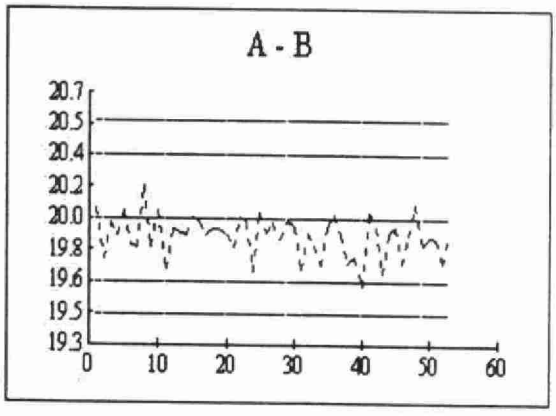
| *Number* | *Expected* | *Mean* | *Std. Dev.* |
|---|---|---|---|
| 53 | 35 | 35.139 | 0.383 |
| 53 | 21 | 21.156 | 0.24 |
| 53 | 7 | 6.987 | 0.099 |

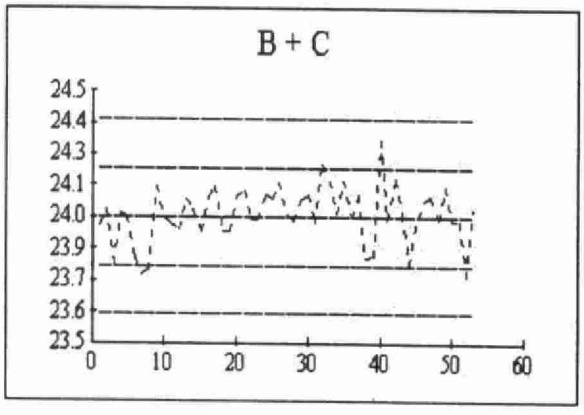
| **OTHER CHECKS** | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 53 | -0.045 | 0.068 |
| Digested Blank | 53 | -0.032 | 0.066 |

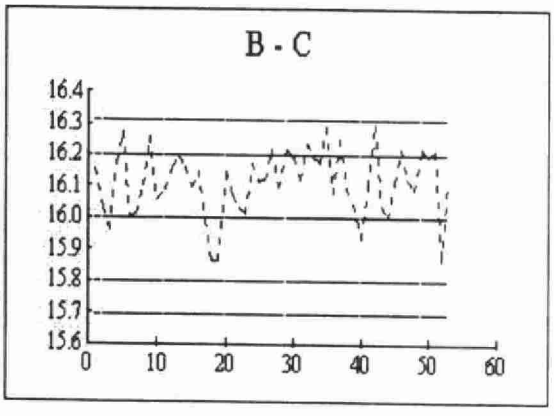
Nitrogen; total Kjeldahl ( E3368A )

QC Data: 1/1/02 to 12/31/02



# OXYGEN DEMAND, BIOCHEMICAL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '61 |
| Method Reference No. | E3182 | Reporting Unit | mg/L as $O_2$ |
| LIMS Product Code | BOD3182 | Supervisor | P. Wilson |
| Sample Type/Matrix | Raw Sewage, Industrial Waste, Effluent, Drinking Water, Ground Water, Leachate, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required: | 400 mL |
| Container: | Glass or plastic |

## SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

## ANALYTICAL PROCEDURE:

Oxygen depletion is measured as the difference in dissolved oxygen (DO) concentration. DO readings are taken prior to sample storage, and also at the end of storage in the dark at 20$^0$C for five days (BOD5). If necessary, dilutions are made with aerated, nutrient-enriched water to obtain a 25-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples, calculation of an appropriate seed correction is required.

## INSTRUMENTATION:

-YSI Model 59 DO meter (Yellow Springs Instrument Company) with DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen (1 mil = 0.001 inch).
-Titration equipment for Winkler analysis of dissolved oxygen.
-Incubator (19-21$^0$C); BOD bottles (300 mL)

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |

## CALIBRATION (DO):

The standard is air-saturated reversed osmosis deionized water. The DO content is read from a table (ORBISPHERE LABORATORIES - Pressure temperature dissolved oxygen table) after measuring its temperature and the barometric pressure in the laboratory.

CONTROLS:

| | |
|---|---|
| Calibration (DO) | 2 QC solutions of Pure-DW water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are compared using the oxygen meter and the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO Analyzer. |
| Recovery (BOD5)* | 3 Recovery standards prepared from a combination of Glucose and Glutamic Acid e.g. R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation. |
| Drift | Air saturated Pure-DW water after every 24 samples. |
| **Blanks*** | Pure-DW water and BOD dilution water |

NOTES:

* These solutions are incubated for five days alongside samples.

## OXYGEN DEMAND, BIOCHEMICAL (E3182)

QUALITY CONTROL DATA FROM 01/02/02 TO 12/24/02

Analytical Range: to 9.0 mg/L as $O_2$ at 20°C

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 89 | 0.00 | 0.0215 | -0.0215 | 0.1087 |
| B: | 89 | 0.00 | 0.0248 | -0.0248 | 0.0931 |

On any given day the calibration is accepted if the values obtained lie within the ranges:

-0.25 - 0.25

**RECOVERIES:**

| Number of Data | Expected Depletion | Mean Depletion | Standard Deviation (1) |
|---|---|---|---|
| 45 | 2.17 | 2.09 | 0.1916 |
| 44 | 4.34 | 4.13 | 0.2128 |
| 45 | 6.54 | 6.24 | 0.2702 |

**DUPLICATES:**

| n Data Pairs | Sample Depletion Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 73 | 0.0 - 1.8 | 0.1678 | 21.1 |
| 18 | 1.9 - 4.5 | 0.1913 | 6.6 |
| 19 | 4.6 - 9.0 | 0.1573 | 2.0 |
| 110 | Overall | 0.1701 | |

**OTHER CHECKS:**

| | n | Mean | Standard Deviation (1) |
|---|---|---|---|
| 5 Day Pure-DW Blank | 45 | 0.161 | 0.1922 |
| 5 Day BOD Blank | 45 | 0.099 | 0.1946 |

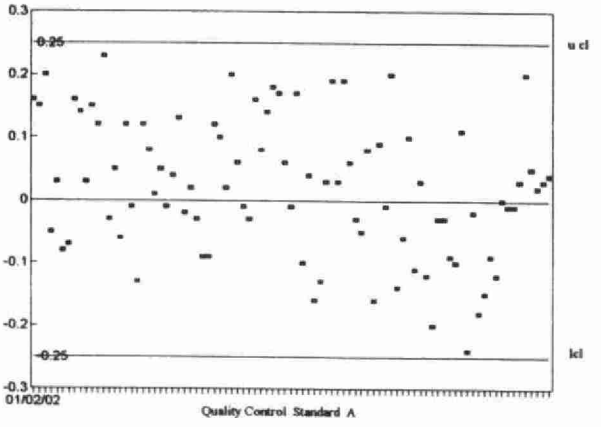
**NOTES:**

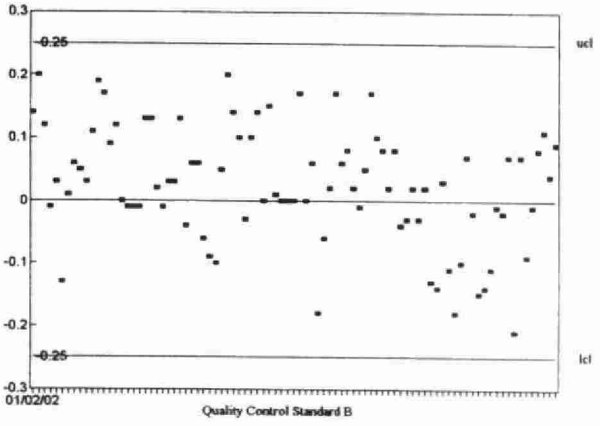
The final concentration of BOD in mg/L as $O_2$ is determined by the oxygen depletion after 5 days at 20°C multiplied by a dilution and seed correction factor.

## OXYGEN DEMAND, BIOCHEMICAL (E3182)

QUALITY CONTROL DATA FROM 01/02/02 TO 12/24/02

Analytical Range: to 9.0 mg/L as $O_2$ at 20°C



# OXYGEN DEMAND, CHEMICAL

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/07/82 |
| Method Reference No. | E3170 | Reporting Unit | mg/L as $O_2$ |
| LIMS Product Code | COD3170 | Supervisor | P. Wilson |
| Sample Type/Matrix | Drinking Water, Ground Water, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 25 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.05 at the full scale level.

**INSTRUMENTATION:**

-Culture tubes with Teflon closures; mechanical-convection oven
-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 1 | Current T value: 5 |

**CALIBRATION:**

3 digested BL plus 3 digested standards

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 digested standards, e.g. QCA |
| Drift | Undigested BL every 10 samples; standard plus BL at end of run |
| Recovery | 2 digested standards, e.g. R1 |
| Interference | Digested standard (40 mg/L as $O_2$) spiked with 50 mg/L Cl confirms suppression of chloride interference. |

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.

The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

## Oxygen Demand Chemical (E3170)

### Analytical Range: to 50 mg/L as $O_2$

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 34 | 40 | 39.589 | -0.411 | 1.182 |
| **B** | 34 | 10 | 9.53 | -0.470 | 1.325 |
| **A + B** | | 50 | 49.119 | -0.881 | 1.989 |
| **A - B** | | 30 | 30.059 | 0.059 | 1.533 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 1.2557 |
| | Within Runs | 1.084 |
| | Between/Within | 1.1584 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 52 | 48 | 53.7 | 46.3 |
| A - B | 32 | 28 | 32.8 | 27.2 |

**DUPLICATES:**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 50 | 0 - 10% | 1.083 | 35.7 |
| 4 | 10 - 20% | 0.530 | 7.9 |
| 4 | 20 - 50% | 1.552 | 10.4 |
| 27 | 50 - 100% | 1.735 | 6.3 |
| 0 | Total | 3.101 | |

**RECOVERIES:**

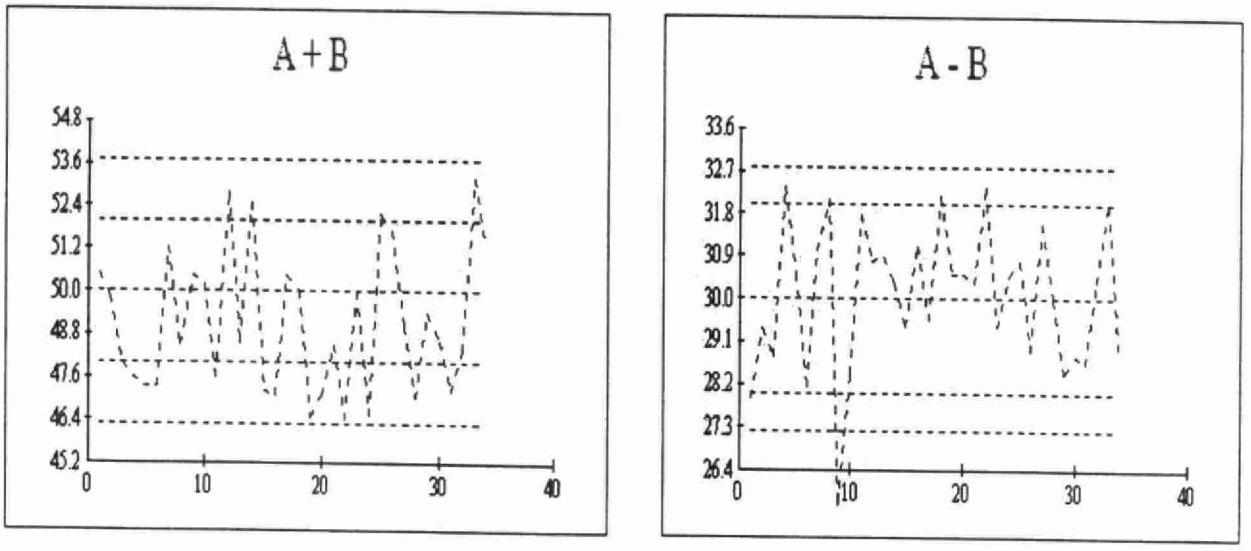
| Number | Expected | Mean | Std. Dev. |
|---|---|---|---|
| 34 | 40 | 36.887 | 2.549 |
| 34 | 10 | 9.09 | 1.489 |

**OTHER CHECKS:**

| | n | Mean | Std Dev. |
|---|---|---|---|
| Chloride Check | 31 | 38.5765 | 2.9424 |
| Digested Blank | 31 | 19.2581 | 7.9623 |

**Oxygen Demand Chemical ( E3170 )**

**QC Data: 1/1/02 to 12/31/02**




Note: For explanation of any exceedence, refer to raw data file.

# OXYGEN DEMAND, CHEMICAL

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/07/82 |
| Method Reference No. | E3246 | Reporting Unit | mg/L as $O_2$ |
| LIMS Product Code | COD3246 | Supervisor | P. Wilson |
| Sample Type/Matrix | Raw Sewage, Industrial Waste, Ground Water, Leachate, Effluent, Sludge, Surface Water, Process Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 25 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.6 at the full scale level.

**INSTRUMENTATION:**

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |

**CALIBRATION:**

2 digested BL plus 4 digested standards

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 digested standards, e.g. QCA |
| Drift | Undigested BL every 10 samples; standard plus BL at end of run |
| Recovery | 2 digested standards, e.g. R1 |
| Interference | Digested standard (50 mg/L as $O_2$) spiked with 900 mg/L Cl confirms suppression of chloride interference. |

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.

The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

## Oxygen Demand Chemical (E3246)

### Analytical Range: to 400 mg/L as $O_2$

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 18 | 400 | 398.811 | -1.189 | 4.867 |
| **B** | 18 | 100 | 103.598 | 3.598 | 3.096 |
| **A + B** | | 500 | 502.409 | 2.409 | 5.613 |
| **A - B** | | 300 | 295.213 | -4.787 | 5.919 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 4.0788 |
| | Within Runs | 4.1854 |
| | Between/Within | 0.9745 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 510 | 490 | 522.5 | 477.5 |
| A - B | 310 | 290 | 315 | 285 |

**DUPLICATES:**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 26 | 0 - 10% | 1.395 | 7.4 |
| 8 | 10 - 20% | 8.388 | 14.6 |
| 4 | 20 - 50% | 10.169 | 9.2 |
| 7 | 50 - 100% | 14.106 | 4 |
| 45 | Total | 11.812 | |

**RECOVERIES:**

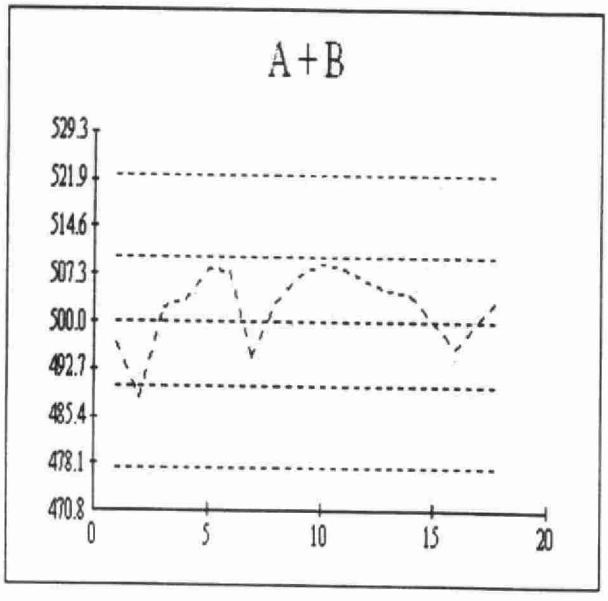
| Number | Expected | Mean | Std. Dev. |
|---|---|---|---|
| 18 | 400 | 386.039 | 11.727 |
| 18 | 100 | 102.62 | 2.867 |

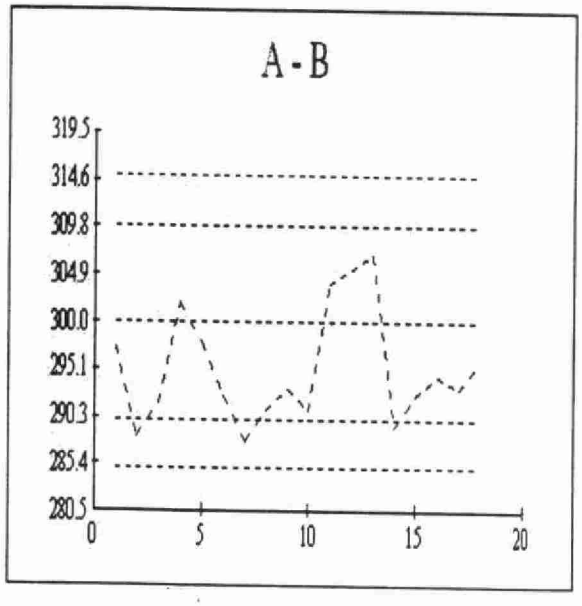
**OTHER CHECKS:**

| | n | Mean | Std Dev. |
|---|---|---|---|
| Chloride Check | 18 | 59.6867 | 6.4002 |
| Digested Blank | 18 | 25.1111 | 3.7281 |

## Oxygen Demand Chemical ( E3246 )

QC Data: 1/1/02 to 12/31/02



# pH

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 09/07/80 |
| Method Reference No | E3218 | Reporting Units | Dimensionless |
| LIMS Product Code | PHALCO3218, CONDPH3218 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or Plastic |

## ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (20.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint alkalinity, and conductivity are determined simultaneously.

## INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

## REPORTING:

| |
|---|
| Maximum Significant Figures: 3 |

## CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

## CONTROLS:

| | |
|---|---|
| Calibration | 2 QC standards e.g. QCA |
| Drift | In run standards throughout the run (diluted tap water 50% V/V) |

## pH (E3218)

QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

Analytical Range: to 14.00 Dimensionless

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 75 | 7.41 | 7.43 | 0.02 | 0.0121 |
| B: | 75 | 4.45 | 4.47 | 0.02 | 0.0524 |
| A+B: | | 11.86 | 11.90 | 0.04 | 0.0562 |
| A-B: | | 2.96 | 2.97 | 0.01 | 0.0596 |

s.d.(AB) S(between runs): 0.038 Sw(within run): 0.042 S/Sw: 0.9

On any given day the calibration is accepted if the values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 11.64 | - | 12.08 | for | A+B |
| 2.79 | - | 3.13 | for | A-B |

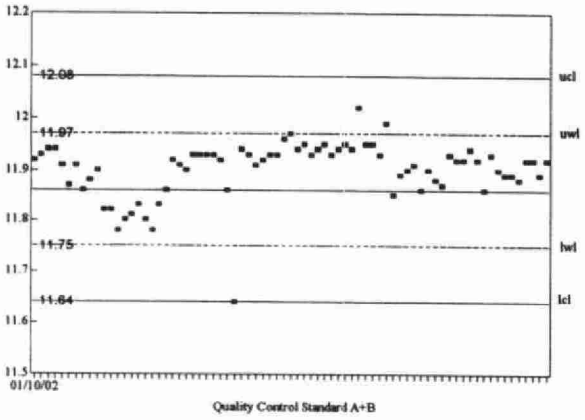
**DUPLICATES:**

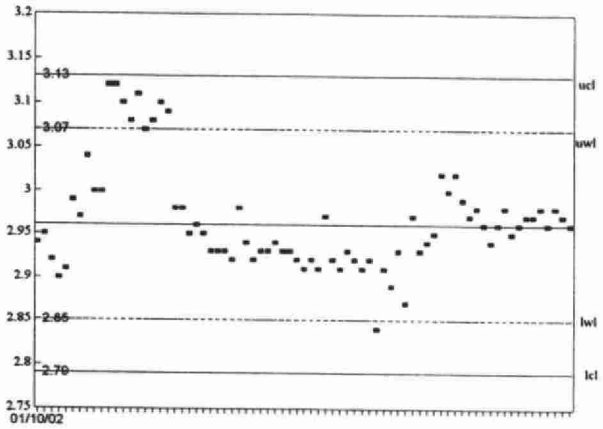
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 27 | 1.00 - 7.00 | 0.0400 | 0.7 |
| 78 | 7.01 - 8.00 | 0.0282 | 0.4 |
| 117 | 8.01 - 12.00 | 0.0338 | 0.4 |
| 222 | Overall | 0.0328 | |

## pH (E3218)

QUALITY CONTROL DATA FROM 01/10/02 TO 12/19/02

Analytical Range: to 14.00 Dimensionless



# PHENOLICS, REACTIVE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/74 |
| Method Reference No. | E3179 | Reporting Unit | µg/L as Phenol |
| LIMS Product Code | PHEN3179 | Supervisor | P.Wilson |
| Sample Type/Matrix | Ground Water, Surface Water, Effluent, Drinking Water, Leachate, Raw Sewage, Industrial Waste, Process Water, Precipitation | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 250 mL |
| Container | Glass, (Phenol bottle with white cap containing preservative is available) |
| Preservative | Sulfuric acid to pH 1.5 - 2 |

## ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide.
Approximate absorbance: 0.03 at the full scale level.

## INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm. Data capture and processing via a Labtronics System.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.2 | Current T value: 1.0 |

## CALIBRATION:

BL plus 2 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 2 standards, e.g. QCA (see note) |
| Drift | BL ,standard ,BL every 10 samples |

## NOTES:

An additional Quality Control Standard (QCC) was added to the method in March 1997.
The HP data capture / processing system was replaced by Labtronics in August 2002.

## Phenolics; 4-AAP (E3179)

### Analytical Range: to 50 ug/L as Phenol

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 32 | 40 | 40.075 | 0.075 | 0.448 |
| **B** | 32 | 10 | 10.302 | 0.302 | 0.223 |
| **C** | 32 | 5 | 5.103 | 0.103 | 0.246 |
| **A + B** | | 50 | 50.377 | 0.377 | 0.578 |
| **A - B** | | 30 | 29.773 | -0.227 | 0.407 |
| **B + C** | | 15 | 15.405 | 0.405 | 0.436 |
| **B - C** | | 5 | 5.199 | 0.199 | 0.173 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.3537 |
| | Within Runs | 0.2878 |
| | Between/Within | 1.229 |
| s.d.(BC) | Between Runs | 0.2345 |
| | Within Runs | 0.1223 |
| | Between/Within | 1.9174 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 51.1 | 48.9 | 52.2 | 47.8 |
| A - B | 31.1 | 28.9 | 31.7 | 28.3 |
| B + C | 15.5 | 14.5 | 16 | 14 |
| B - C | 5.5 | 4.5 | 5.75 | 4.25 |

**DUPLICATES:**

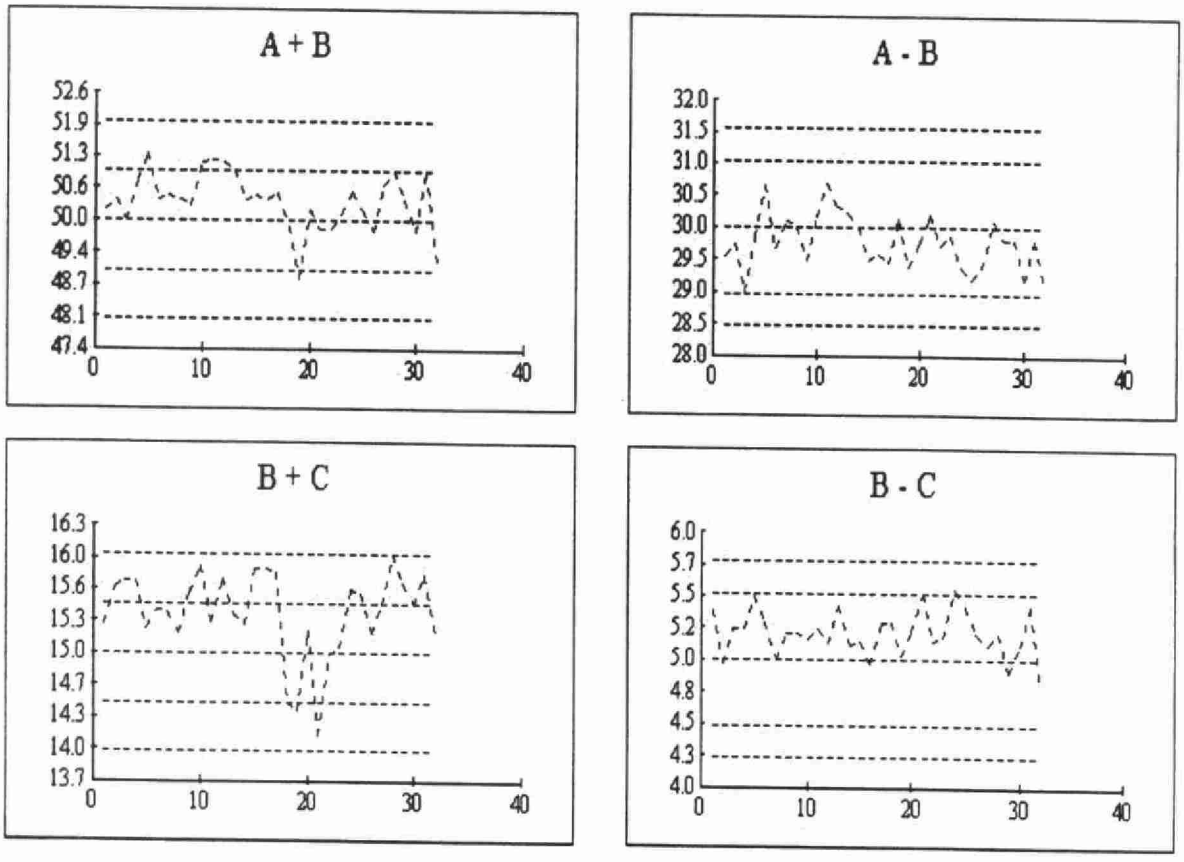
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 65 | 0 - 10% | 0.163 | 49 |
| 1 | 10 - 20% | N.A. | N.A. |
| 2 | 20 - 50% | N.A. | N.A. |
| 0 | 50 - 100% | N.A. | N.A. |
| 68 | Total | 0.167 | |

**OTHER CHECKS:**

| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 28 | 0.015 | 0.183 |

Phenolics; 4-AAP [ E3179 ]

QC Data; 1/1/02 to 12/31/02



## PHOSPHOROUS, REACTIVE ortho-PHOSPHATE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No. | E3364 | Reporting Unit | mg/L as P |
| LIMS Product Code | DISNUT3364 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
Approximate absorbance: 0.2 at the full scale level.
Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.
Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.0005 | Current T value: 0.0025 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL ,standard ,and BL after every 10 samples |

**NOTES:**

The HP data capture / processing system was replaced by Labtronics in August 1999.

**Phosphorus; reactive ortho-phosphate phosphate (E3364)**

**Analytical Range: to 0.100 mg/L as P**

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 86 | 0.08 | 0.0798 | -0.0002 | 0.0008 |
| **B** | 86 | 0.04 | 0.0402 | 0.0002 | 0.0009 |
| **C** | 86 | 0.008 | 0.0081 | 0.0001 | 0.0006 |
| **A + B** | | 0.12 | 0.1201 | 0.0001 | 0.0012 |
| **A - B** | | 0.04 | 0.0396 | -0.0004 | 0.0012 |
| **B + C** | | 0.048 | 0.0484 | 0.0004 | 0.0012 |
| **B - C** | | 0.032 | 0.0321 | 0.0001 | 0.0009 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0008 |
| | Within Runs | 0.0008 |
| | Between/Within | 1 |
| s.d.(BC) | Between Runs | 0.0008 |
| | Within Runs | 0.0006 |
| | Between/Within | 1.3333 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 0.1224 | 0.1176 | 0.1248 | 0.1152 |
| A - B | 0.0424 | 0.0376 | 0.0436 | 0.0364 |
| B + C | 0.0496 | 0.0464 | 0.0512 | 0.0448 |
| B - C | 0.0332 | 0.0302 | 0.0344 | 0.0296 |

**DUPLICATES**

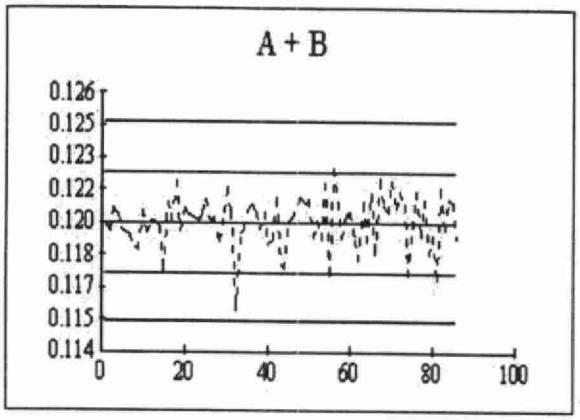
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 193 | 0 - 10% | 0.0009 | 29.5 |
| 20 | 10 - 20% | 0.0011 | 7.8 |
| 16 | 20 - 50% | 0.0018 | 6 |
| 6 | 50 - 100% | 0.0011 | 1.6 |
| 235 | Total | 0.001 | |

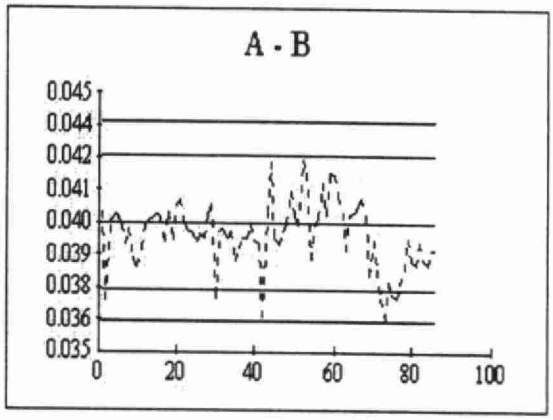
**OTHER CHECKS**

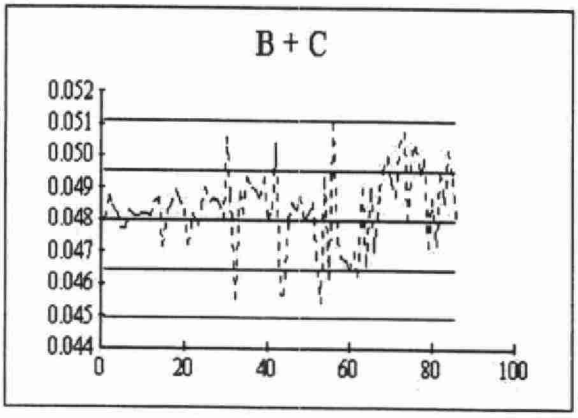
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 86 | 0.001 | 0.001 |

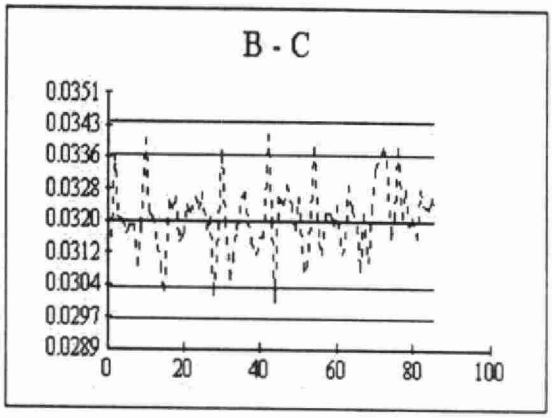
Phosphorus; phosphate ( E3364A )

QC Data: 1/1/02 to 12/31/02



# PHOSPHORUS, REACTIVE ortho-PHOSPHATE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No | E3366 | Reporting Unit | mg/L as P |
| LIMS Product Code | DISNUT3366 | Supervisor | P.Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Ground Water. | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
Approximate absorbance: 0.5 at the full scale level.
Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.
Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL ,standard and BL every 10 samples |

## NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

**Phosphorus; reactive ortho-phosphate (E3366)**

**Analytical Range: to 10.0 mg/L as P**

**CALIBRATION CONTROL**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 55 | 8 | 7.98 | -0.02 | 0.045 |
| **B** | 55 | 4 | 4.014 | 0.014 | 0.029 |
| **C** | 55 | 0.8 | 0.802 | 0.002 | 0.015 |
| **A + B** | | 12 | 11.994 | -0.006 | 0.058 |
| **A - B** | | 4 | 3.967 | -0.033 | 0.05 |
| **B + C** | | 4.8 | 4.816 | 0.016 | 0.037 |
| **B - C** | | 3.2 | 3.212 | 0.012 | 0.027 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.038 |
| | Within Runs | 0.0354 |
| | Between/Within | 1.0734 |
| s.d.(BC) | Between Runs | 0.0231 |
| | Within Runs | 0.0191 |
| | Between/Within | 1.2094 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 12.14 | 11.86 | 12.29 | 11.71 |
| A - B | 4.14 | 3.86 | 4.22 | 3.78 |
| B + C | 4.87 | 4.73 | 4.94 | 4.66 |
| B - C | 3.27 | 3.13 | 3.31 | 3.09 |

**DUPLICATES**

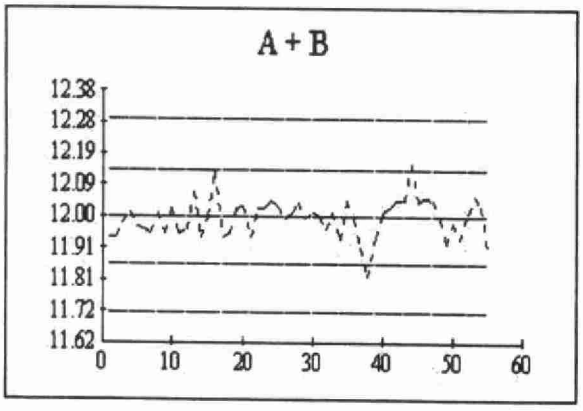
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 109 | 0 - 10% | 0.021 | 17.4 |
| 9 | 10 - 20% | 0.019 | 1.4 |
| 7 | 20 - 50% | 0.114 | 2.9 |
| 6 | 50 - 100% | 0.064 | 0.9 |
| 131 | Total | 0.035 | |

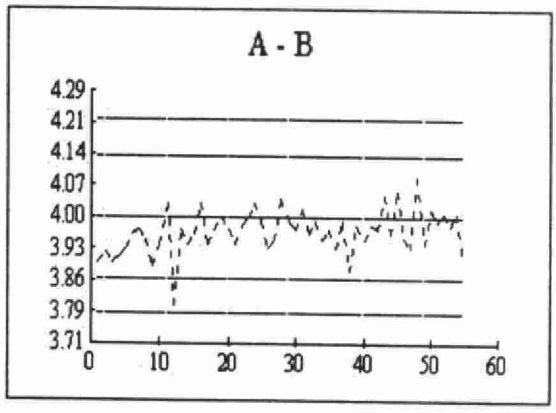
**OTHER CHECKS**

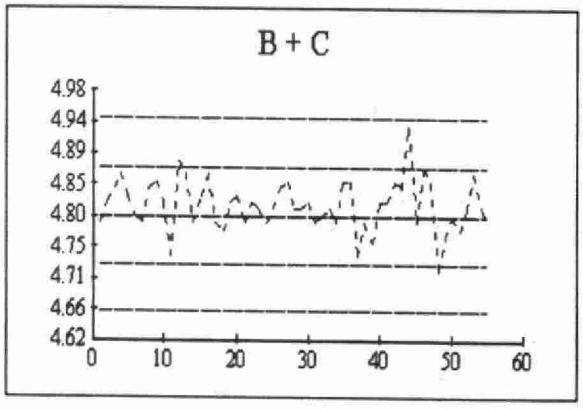
| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 54 | -0.014 | 0.008 |

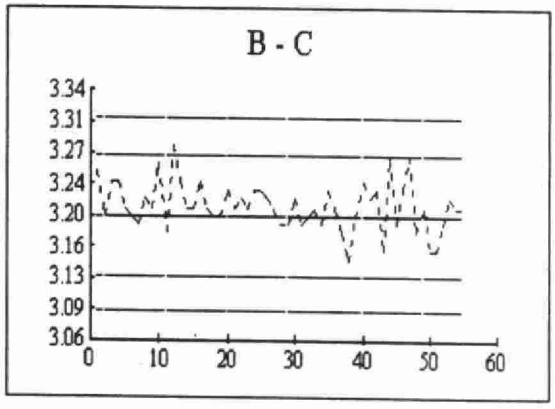
Phosphorus; phosphate ( E3366A )

QC Data; 1/1/02 to 12/31/02



# PHOSPHORUS, TOTAL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Mar '89 |
| Method Reference No. | E3116 | Reporting Unit | mg/g as P |
| LIMS Product Code | TNP3116 | Supervisor | P. Wilson |
| Sample Type/Matrix | Soil, Sediment, Dried Sludge | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 0.08 to 0.4 g |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

## INSTRUMENTATION:

Hot plate
Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.
Data capture and processing via a computer system

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

## CALIBRATION:

3 High and 2 Low Calibration Standards

## CONTROLS:

| | |
|---|---|
| Calibration | In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92) |
| Drift | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery | 1 digested BL plus 4 digested standards |

## NOTES:

System is calibrated with undigested standards.

## PHOSPHORUS, TOTAL (E3116)

QUALITY CONTROL DATA FROM 01/29/02 TO 12/24/02

Analytical Range: to 2 mg/L as P

**QUALITY CONTROL:**

| | n | Expected Concentration (mg/g) | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| RS92 In House Soil Composite | 202 | 0.47 | 0.475 | 0.005 | 0.0255 |
| RSM-2781 - Domestic Sludge (non certified) | 0 | 24.0 | 24.7 | 0.7 | 0.8763 |

The run is accepted if the control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 0.4 | - | 0.54 | for | RS92 |
| 17.9 | - | 30.1 | for | RSM-2781 |

**Recovery Standards**

| | n | Expected Concentration (mg/L) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| R1 | 20 | 1.05 | 1.070 | 0.0181 |
| R2 | 20 | 0.35 | 0.371 | 0.0168 |

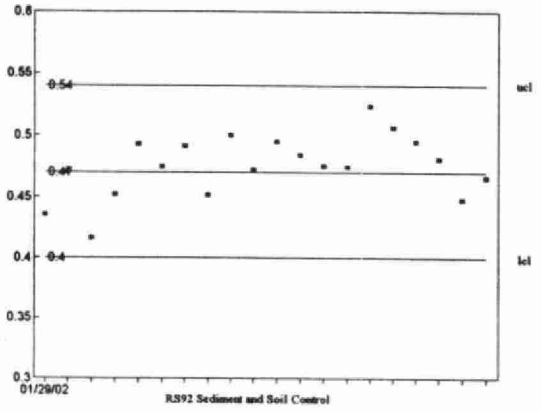
**DUPLICATES: (Sediment/Soils)**

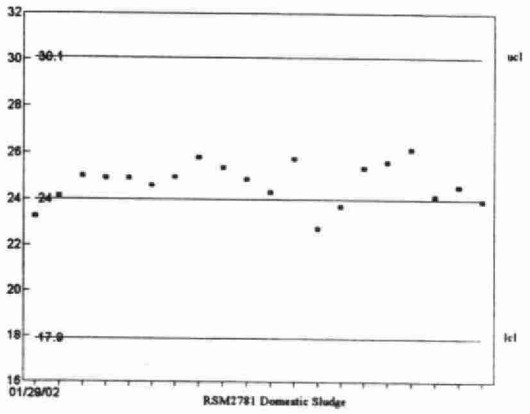
| n Data Pairs | Sample Concentration Span (mg/g) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 6 | 0.00 - 0.50 | 0.0371 | 9.7 |
| 32 | 0.51 - 1.00 | 0.0467 | 6.0 |
| 16 | 1.00 - 2.50 | 0.0392 | 2.7 |
| 0 | 2.51 - 5.00 | N.A. | N.A. |
| 54 | Overall | 0.0436 | |

## PHOSPHORUS, TOTAL (E3116)

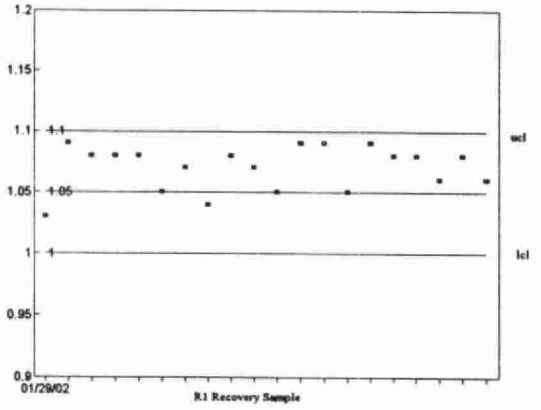
QUALITY CONTROL DATA FROM 01/29/02 TO 12/04/02

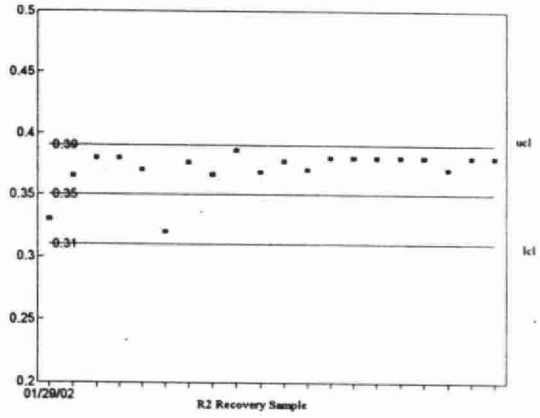
### Analytical Range: to 2 mg/g as P




### Analytical Range: to 2 mg/L as P



# PHOSPHORUS, TOTAL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Mar '89 |
| Method Reference No. | E3118 | Reporting Unit | mg/g as P |
| LIMS Product Code | TNP3118 | Supervisor | P. WILSON |
| Sample Type/Matrix | Vegetation, Moss Bag | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 0.02 to 0.04 g |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

## INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

## CALIBRATION:

3 High and 2 Low Calibration Standards

## CONTROLS:

| | |
|---|---|
| Calibration | In house composite A-VEG, plus QC VEG (Pine Needles) |
| Drift | 4 BL's per run; high and low calibration standard at the end of the run |
| Recovery | 1 digested BL plus 4 digested standards |

## NOTES:

System is calibrated with undigested standards.

## PHOSPHORUS, TOTAL (E3118)

QUALITY CONTROL DATA FROM 03/05/02 TO 12/04/02

Analytical Range: to 2 mg/L as P

### CALIBRATION CONTROL:

| | n | Expected Concentration (mg/g) | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| Pine Needles | 7 | 1.2 | 1.147 | -0.0529 | 0.0451 |

The calibration is accepted if the calibration control values obtained lie within the ranges:
1.1 - 1.4 for pine needles

### Recovery Standards

| | n | Expected Concentration (mg/L) | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| R1 | 7 | 1.05 | 1.070 | 0.0163 |
| R2 | 7 | 0.35 | 0.378 | 0.0064 |

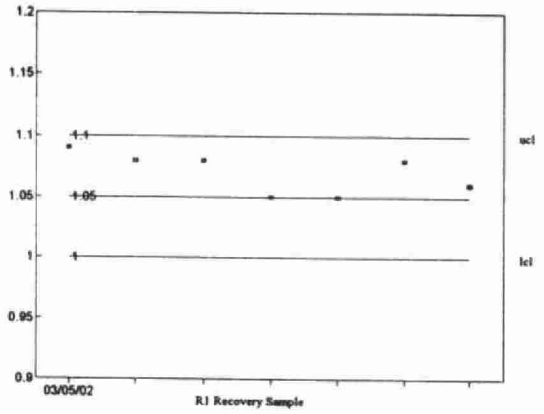
### DUPLICATES: (VEGETATION)

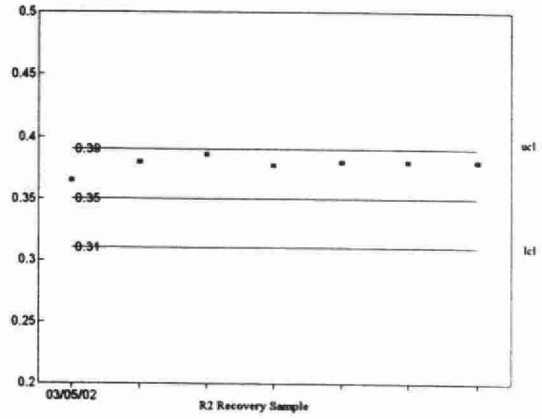
| n Data Pairs | Sample Concentration Span (mg/g) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 1 | 0.00 - 0.50 | N.A. | N.A. |
| 4 | 0.51 - 2.50 | 0.0828 | 4.3 |
| 13 | 2.51 - 5.00 | 0.1489 | 3.9 |
| 18 | Overall | 0.1326 | |

## PHOSPHORUS, TOTAL (E3118)

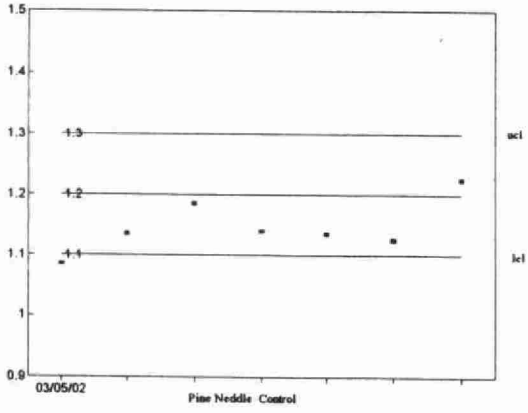
QUALITY CONTROL DATA FROM 03/05/02 TO 12/04/02

Analytical Range: to 2 mg/L as P




Analytical Range: to 2 mg/g as P



# PHOSPHORUS, TOTAL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No | E3367 | Reporting Unit | mg/L as P |
| LIMS Product Code | TOTNUT3367 | Supervisor | P.Wilson |
| Sample Type/Matrix | Precipitation, Drinking Water, Surface Water | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180$^0$C, 210$^0$C and 360$^0$C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level.

Total Kjeldahl nitrogen is determined simultaneously.

## INSTRUMENTATION:

Three Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture and processing via a computer system

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.002 | Current T value: 0.010 |

## CALIBRATION:

BL plus 7 undigested standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 undigested standards, e.g. QCA |
| Drift | BL , undigested standard , BL every 10 samples |
| Recovery | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

## NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999.

## Phosphorus; Total (E3367)

### Analytical Range: to 0.200 mg/L as P

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| A | 80 | 0.16 | 0.159 | -0.001 | 0.0012 |
| B | 80 | 0.08 | 0.08 | 0.000 | 0.0007 |
| C | 80 | 0.016 | 0.016 | 0.000 | 0.0005 |
| A + B | | 0.24 | 0.239 | -0.001 | 0.0016 |
| A - B | | 0.08 | 0.08 | 0.000 | 0.0012 |
| B + C | | 0.096 | 0.095 | -0.001 | 0.0010 |
| B - C | | 0.064 | 0.064 | 0.000 | 0.0007 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.001 |
| | Within Runs | 0.0008 |
| | Between/Within | 1.25 |
| s.d.(BC) | Between Runs | 0.0006 |
| | Within Runs | 0.0005 |
| | Between/Within | 1.2 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 0.2434 | 0.2366 | 0.2468 | 0.2332 |
| A - B | 0.0834 | 0.0766 | 0.0851 | 0.0749 |
| B + C | 0.098 | 0.094 | 0.1 | 0.092 |
| B - C | 0.066 | 0.062 | 0.067 | 0.061 |

**DUPLICATES**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 170 | 0 - 10% | 0.0019 | 20.6 |
| 35 | 10 - 20% | 0.0011 | 4 |
| 21 | 20 - 50% | 0.002 | 3.9 |
| 3 | 50 - 100% | 0.0012 | 0.8 |
| 229 | Total | 0.0018 | 10.2 |

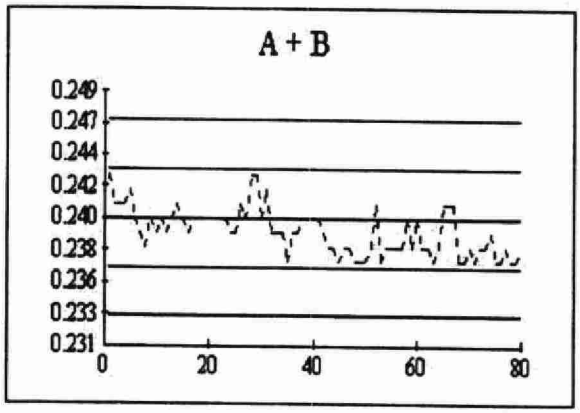
**RECOVERIES**

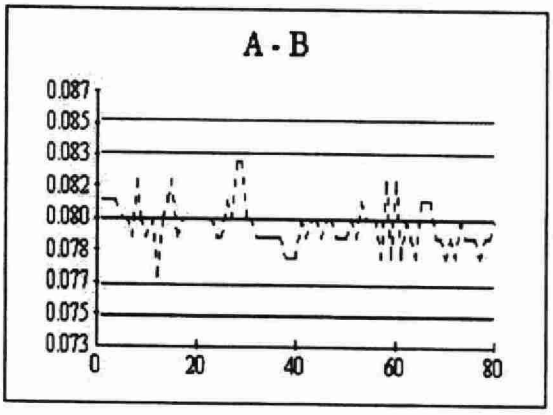
| *Number* | *Expected* | *Mean* | *Std. Dev.* |
|---|---|---|---|
| 80 | 0.14 | 0.138 | 0.002 |
| 80 | 0.084 | 0.083 | 0.001 |
| 80 | 0.028 | 0.029 | 0.001 |

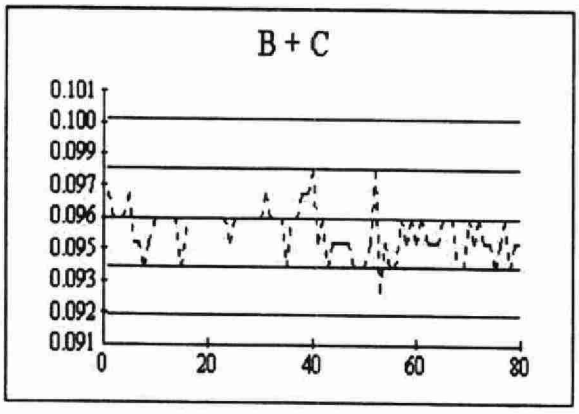
| OTHER CHECKS | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 80 | 0.00001 | 0.0005 |
| Digested Blank | 80 | 0.001 | 0.001 |

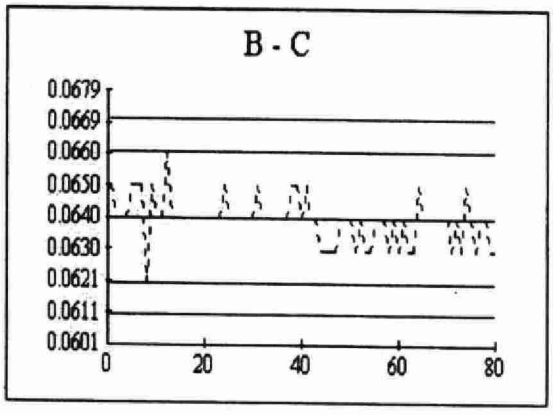
Phosphorus; total [ E3367A ]

QC Data: 01/01/02 to 12/31/02



# PHOSPHORUS, TOTAL

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/04/79 |
| Method Reference No. | E3368 | Reporting Unit | mg/L as P |
| LIMS Product Code | TOTNUT3368 | Supervisor | J. McBride |
| Sample Type/Matrix | Sludge, Raw Sewage, Industrial Waste, Effluent, Ground Water, Process Water, Leachate. | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

## ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
Approximate absorbance: 0.8 at the full scale level.
Total Kjeldahl Nitrogen is determined simultaneously.

## INSTRUMENTATION:

3-Block digesters
Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using an IR sensitive phototube. Data capture and processing via a computer system.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | BL every 10 samples; undigested standard every 20 samples |
| Recovery | 3 digested BL plus 3 digested standards in duplicate, e.g. R1 |

## NOTES:

System is calibrated with undigested standards.
The HP capture / processing system was replaced by Labtronics in April 1999

## Phosphorus; total (E3368)

### Analytical Range: to 10.0 mg/L as P

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| A | 52 | 8 | 7.986 | -0.014 | 0.032 |
| B | 52 | 4 | 3.994 | -0.006 | 0.017 |
| C | 52 | 0.8 | 0.797 | -0.003 | 0.007 |
| A + B | | 12 | 11.979 | -0.021 | 0.041 |
| A - B | | 4 | 3.992 | -0.008 | 0.032 |
| B + C | | 4.8 | 4.791 | -0.009 | 0.02 |
| B - C | | 3.2 | 3.196 | -0.004 | 0.017 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.0259 |
| | Within Runs | 0.0226 |
| | Between/Within | 1.146 |
| s.d.(BC) | Between Runs | 0.0129 |
| | Within Runs | 0.012 |
| | Between/Within | 1.075 |

**CONTROL LIMITS**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 12.065 | 11.935 | 12.13 | 11.87 |
| A - B | 4.065 | 3.935 | 4.097 | 3.903 |
| B + C | 4.834 | 4.766 | 4.868 | 4.732 |
| B - C | 3.234 | 3.166 | 3.251 | 3.149 |

**DUPLICATES**

| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 124 | 0 - 10% | 0.047 | 22.7 |
| 12 | 10 - 20% | 0.035 | 2.6 |
| 4 | 20 - 50% | 0.025 | 0.9 |
| 0 | 50 - 100% | N.A. | N.A. |
| 140 | Total | 0.045 | 12 |

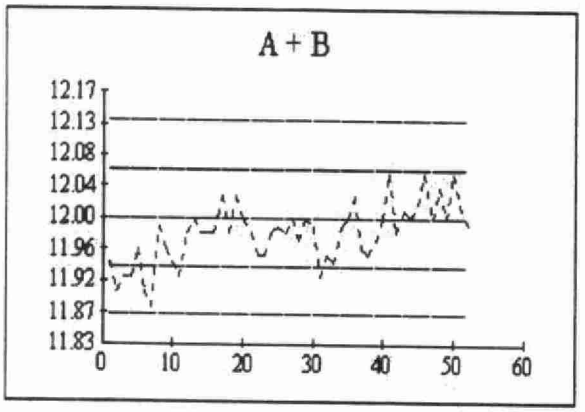
**RECOVERIES**

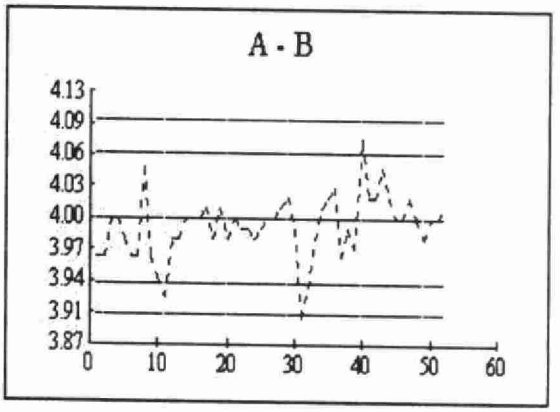
| *Number* | *Expected* | *Mean* | *Std. Dev.* |
|---|---|---|---|
| 52 | 7 | 6.953 | 0.083 |
| 52 | 4.2 | 4.191 | 0.049 |
| 52 | 1.4 | 1.404 | 0.019 |

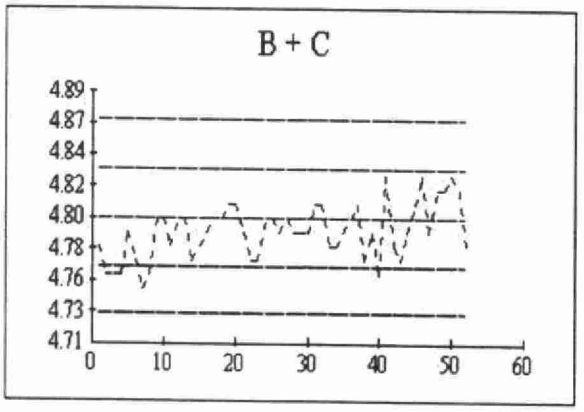
| OTHER CHECKS | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 52 | 0.007 | 0.01 |
| Digested Blank | 52 | 0.008 | 0.009 |

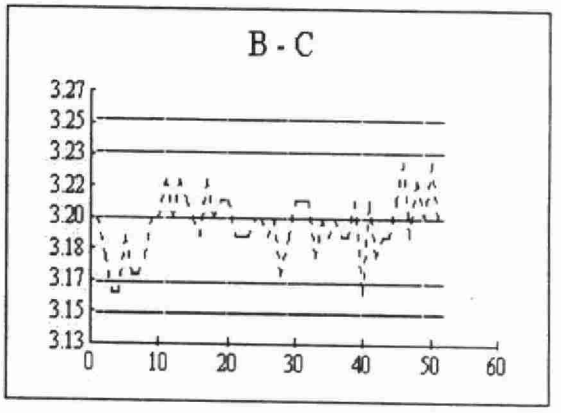
Phosphorus; total [ E3368A ]

QC Data; 1/1/02 to 12/31/02



# SILICON, REACTIVE SILICATES

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 01/02/75 |
| Method Reference No. | E3370 | Reporting Unit | mg/L as Si |
| LIMS Product Code | DCSI3370 | Supervisor | P.Wilson |
| Sample Type/Matrix | Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 10 mL |
| Container | Plastic |

**ANALYTICAL PROCEDURE:**

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.
Approximate absorbance: 0.7 at the full scale level.
Dissolved inorganic and dissolved organic carbon are determined simultaneously.

**INSTRUMENTATION:**

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 660 nm. Data capture and processing via a computer system.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.02 | Current T value: 0.10 |

**CALIBRATION:**

BL plus 7 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g., QCA |
| Drift | BL, standard and BL every 10 samples. |

**NOTES:**

December 1998: The HP data capture/processing system was replaced by Labtronics.

**Silicon; reactive silicates (E3370)**

**Analytical Range: to 10.0 mg/L as Si**

**CALIBRATION CONTROL:**

| | Number | Expected | Mean | Mean Bias | Std. Dev. |
|---|---|---|---|---|---|
| **A** | 48 | 8 | 7.992 | -0.008 | 0.034 |
| **B** | 48 | 2 | 1.992 | -0.008 | 0.025 |
| **C** | 48 | 0.5 | 0.468 | -0.032 | 0.031 |
| **A + B** | | 10 | 9.984 | -0.016 | 0.048 |
| **A - B** | | 6 | 6 | 0 | 0.036 |
| **B + C** | | 2.5 | 2.46 | -0.04 | 0.055 |
| **B - C** | | 1.5 | 1.524 | 0.024 | 0.016 |

Between Run VS Within Run Standard Deviations

| | | |
|---|---|---|
| s.d.(AB) | Between Runs | 0.03 |
| | Within Runs | 0.0255 |
| | Between/Within | 1.1765 |
| s.d.(BC) | Between Runs | 0.0284 |
| | Within Runs | 0.0113 |
| | Between/Within | 2.5133 |

**CONTROL LIMITS:**

| Control Standard | Warning Limits | | Control Limits | |
|---|---|---|---|---|
| | Upper | Lower | Upper | Lower |
| A + B | 10.17 | 9.83 | 10.34 | 9.66 |
| A - B | 6.17 | 5.83 | 6.25 | 5.75 |
| B + C | 2.57 | 2.43 | 2.63 | 2.37 |
| B - C | 1.57 | 1.43 | 1.6 | 1.4 |

**DUPLICATES:**

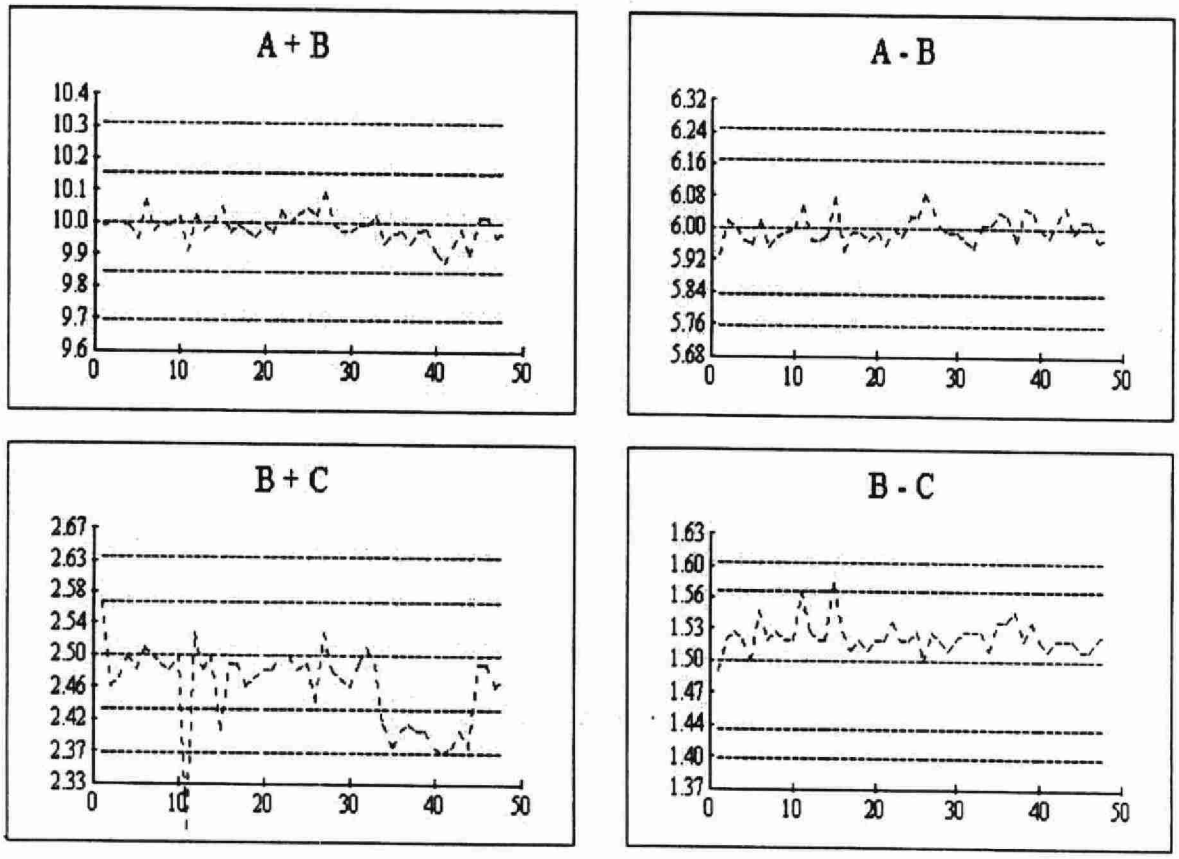
| Number | Conc. Span | Std. Dev. | % Coeff of Var |
|---|---|---|---|
| 71 | 0 - 10% | 0.007 | 1.6 |
| 36 | 10 - 20% | 0.014 | 1 |
| 27 | 20 - 50% | 0.12 | 3.9 |
| 8 | 50 - 100% | 0.025 | 0.4 |
| 142 | Total | 0.053 | 3.4 |

**OTHER CHECKS:**

| | Number | Mean | Std. Dev. |
|---|---|---|---|
| LTB | 48 | -0.041 | 0.027 |

Silicon; reactive silicate [E3370A ]

QC Data: 1/1/02 to 12/31/02




Note: For explanation of any exceedence, refer to raw data file.

## SOLIDS, DISSOLVED

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '61 |
| Method Reference No. | E3188 | Reporting Unit | mg/L |
| LIMS Product Code | TSD3188,DS3188,DIGN3188 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 125 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Sample is filtered under moderate suction through a Whatman 934AH grade glass fibre filter. Generally 100 mL of filtrate (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103±2$^{0}$C, and stored in a desiccator for at least 24 hours. The dissolved solids content is calculated by subtracting the original dish mass from the dried residue + dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a computer system.

**INSTRUMENTATION:**

Balance (5 decimal places), drying oven, suction filtration apparatus, dishes (Teflon).
Computer system with appropriate software.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 2 | Current T value: 10 |

**CALIBRATION:**

Balance zero
Balance internal calibration is performed daily.

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams) |
| Drift | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery | 2 standards, e.g. R1 |
| Method Blank | 100 mL Pure Water. |

## SOLIDS, DISSOLVED (E3188)

QUALITY CONTROL DATA FROM 01/02/02 TO 07/26/02

**CALIBRATION CONTROL:**

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 35 | 50.00 | 50.0006 | 0.0006 | 0.00004 |
| B: | 35 | 30.00 | 30.0004 | 0.0004 | 0.00005 |
| A+B: | | 80.00 | 80.0009 | 0.0009 | 0.00007 |
| A-B: | | 20.00 | 20.0002 | 0.0002 | 0.00005 |

s.d.(AB) S(between runs): 0.0004 Sw(within run): 0.00004 S/Sw: 1.15

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0007 - 80.0011 for A+B
20 - 20.0004 for A-B

QUALITY CONTROL DATA FROM 08/21/02 TO 12/17/02

**CALIBRATION CONTROL:**

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 31 | 50.00 | 50.0002 | 0.0002 | 0.00011 |
| B: | 31 | 30.00 | 30.0002 | 0.0002 | 0.00037 |
| A+B: | | 80.00 | 80.0004 | 0.0004 | 0.00041 |
| A-B: | | 20.00 | 20.0000 | 0.0000 | 0.00033 |

s.d.(AB) S(between runs): 0.0003 Sw(within run): 0.0002 S/Sw: 1.17

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0017 - 79.9991 for A+B
20.0001 - 19.999 for A-B

**RECOVERIES:**

| Number of Data | Expected Concentration (mg/L) | Mean Concentration Measured (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| 66 | 2000.0 | 1995.46 | 12.18 |
| 66 | 500.0 | 492.85 | 7.91 |

**DUPLICATES:**

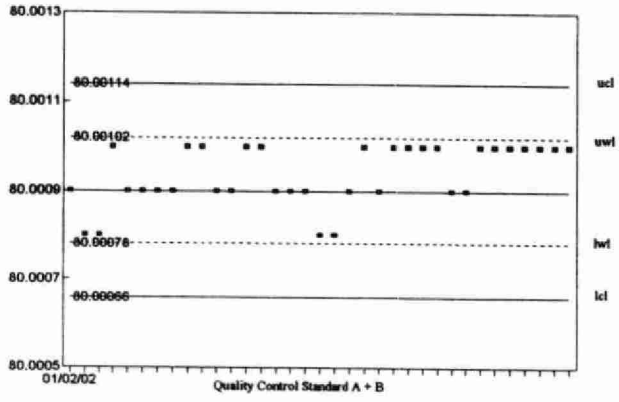
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 27 | 0 - 500 | 12.8150 | 4.1 |
| 93 | 501 - 1000 | 56.4530 | 8.5 |
| 15 | 1001 - 5000 | 53.5835 | 3.3 |
| 135 | Overall | 50.4709 | |

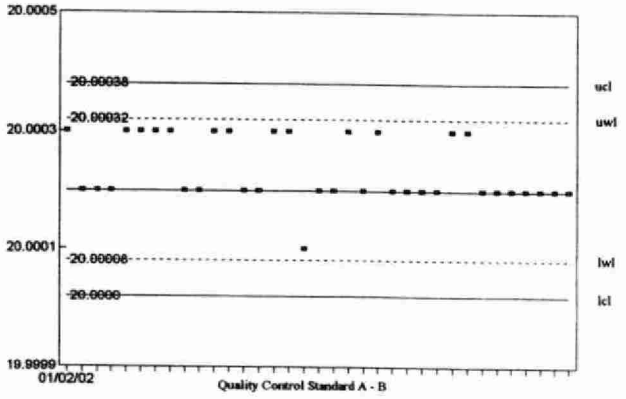
**OTHER CHECKS:**

| | n | Data Mean (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| Blank | 6 | -1.6352 | 9.2703 |

## DISSOLVED, SOLIDS (E3188)

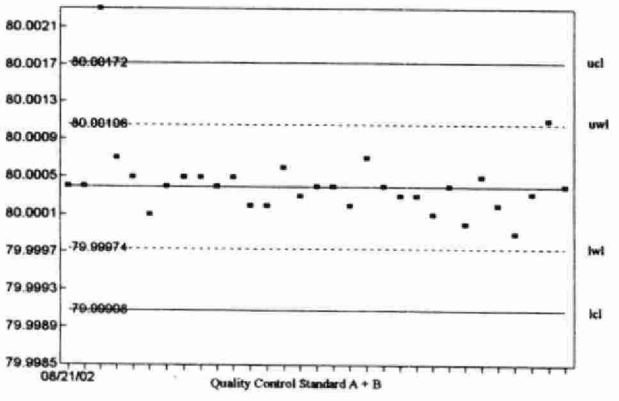
QUALITY CONTROL DATA FROM 01/02/02 TO 07/26/02

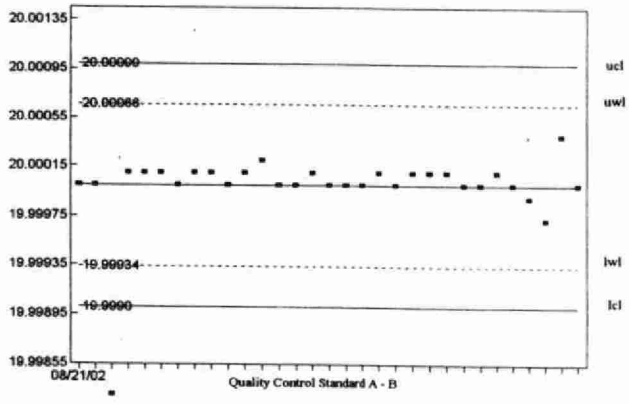



## DISSOLVED, SOLIDS (E3188)

QUALITY CONTROL DATA FROM 08/21/02 TO 12/17/02



# SOLIDS, SUSPENDED

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '81 |
| Method Reference No. | E3188 | Reporting Unit | mg/L |
| LIMS Product Code | TSD3188, SS3188 | Supervisor | P.Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 5-500 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

An appropriately shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 50 mL distilled water. The filter is dried at 103-105$^{0}$C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIMS are controlled by a computer system.

**INSTRUMENTATION:**

Balance (5-decimal places), drying oven, suction filtration apparatus.
Computer system with appropriate software.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams) |
| Drift | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery | 2 standards, e.g. R1 |
| Method Blank | Filter washed with 500 mL distilled water |

**NOTES:**

A standard correction factor (-0.00022g) was applied to all filters to account for weight loss during filtering.
A new set of Q.C. weights was introduced for the year 2000, along with new limits for the weights.

## SOLIDS, SUSPENDED (E3188)

QUALITY CONTROL DATA FROM 01/02/02 TO 08/08/02

### CALIBRATION CONTROL:

Quality Control Data from 01/02/02 to 08/08/02

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| C: | 132 | 0.50 | 0.49990 | -0.00010 | 0.00001 |
| D: | 132 | 0.05 | 0.04994 | -0.00006 | 0.00006 |
| C+D: | | 0.55 | 0.54984 | -0.00016 | 0.00005 |
| C-D: | | 0.45 | 0.44996 | -0.00004 | 0.00006 |

s.d.(CD) S(between runs): 0.00004 Sw(within run): 0.00004 S/Sw: 0.98

The calibration is accepted if the calibration control values (mean mass measured) obtained within the ranges expressed in grams:

0.55007 - 0.54961 for C+D
0.45013 - 0.44979 for C-D

Quality Control Data from 08/14/02 to 12/17/02

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| C: | 129 | 0.50 | 0.50000 | 0.00000 | 0.00001 |
| D: | 129 | 0.05 | 0.05000 | 0.00000 | 0.00001 |
| C+D: | | 0.55 | 0.55001 | 0.00001 | 0.00002 |
| C-D: | | 0.45 | 0.45000 | 0.00000 | 0.00001 |

s.d.(CD) S(between runs): 0.00001 Sw(within run): 0,00001 S/Sw: 1.34

The calibration is accepted if the calibration control values (mean mass measured) obtained within the ranges expressed in grams:

0.550046 - 0.54992 for C+D
0.450027 - 0.44997 for C-D

**RECOVERIES:**

| Number of Data | Expected Concentration (mg/L) | Mean Concentration Measured (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| 261 | 200.0 | 195.15 | 2.3162 |
| 261 | 50.0 | 48.74 | 3.2347 |

**DUPLICATES:**

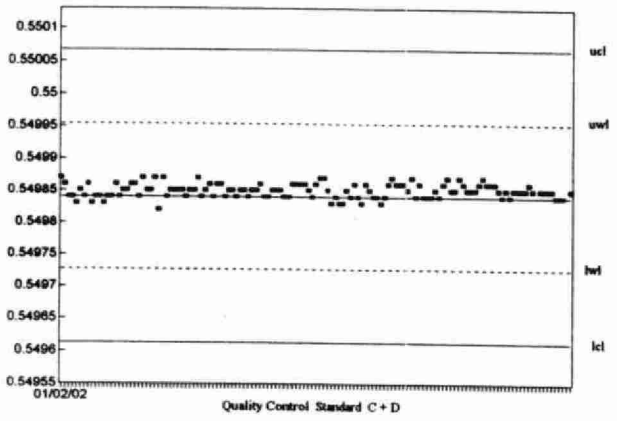
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 265 | 0 - 5 | 0.3450 | 15.9 |
| 120 | 6 - 10 | 0.6018 | 8.5 |
| 137 | 11 - 25 | 0.8475 | 5.6 |
| 99 | 26 - 100 | 1.7544 | 3.7 |
| 33 | 101 - 500 | 4.7719 | 2.7 |
| 7 | 501 - 1000 | 8.4990 | 1.3 |
| 15 | 1001 - 10000 | 74.0874 | 2.3 |
| 676 | Overall | 11.1524 | |

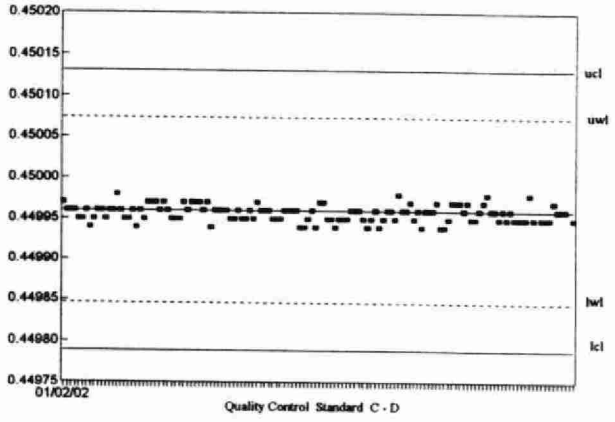
**OTHER CHECKS:**

| | n | Data Mean (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| Blank | 261 | -0.10594 | 0.2296 |

## SOLIDS, SUSPENDED (E3188)

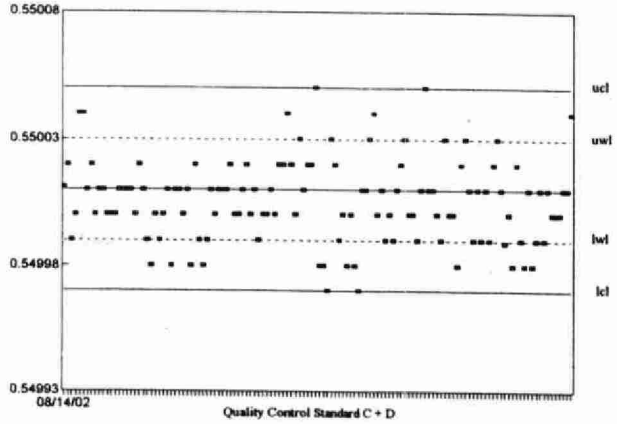
QUALITY CONTROL DATA FROM 01/02/02 TO 08/08/02

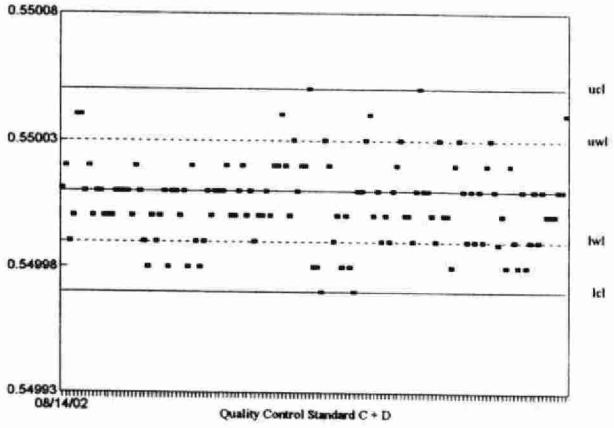



## SOLIDS, SUSPENDED (E3188)

QUALITY CONTROL DATA FROM 08/14/02 TO 12/17/02



## SOLIDS, SUSPENDED IGNITED
## (Particulate Ash and Particulate Loss On Ignition)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '61 |
| Method Reference No. | E3188 | Reporting Unit | mg/L |
| LIMS Product Code | SIGN3188 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 5-500 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

The procedure for particulate solids (SS3188) is followed and the dried residue is ignited at 600±50°C for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The particulate ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for SS3188. The particulate loss on ignition (estimate of volatile suspended solids) is the difference between the final ignited mass plus filter and the residue (suspended solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a computer system.

**INSTRUMENTATION:**

Balance (5 decimal places), muffle furnace, filters, Petri dishes
Computer system with appropriate software

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams) |
| Drift | Balance is reset to zero after every 10 weighings by the microcomputer. |

**SOLIDS, SUSPENDED IGNITED (E3188)**
**(Particulate Ash and Particulate Loss On Ignition)**

QUALITY CONTROL DATA FROM 01/02/02 TO 11/18/02

**CALIBRATION CONTROL:**

QUALITY CONTROL DATA FROM 01/02/02 TO 07/26/02

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| C: | 7 | 0.50 | 0.49990 | -0.00010 | 0.000005 |
| D: | 7 | 0.05 | 0.04995 | -0.00005 | 0.000009 |
| C+D: | | 0.55 | 0.54985 | -0.00015 | 0.000007 |
| C-D: | | 0.45 | 0.44995 | -0.00005 | 0.000013 |

s.d.(CD) S(between runs): 0.00001 Sw(within run): 0.00001 S/Sw: 0.81

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

0.5499 - 0.5498 for C+D
0.44999 - 0.44991 for C-D

**CALIBRATION CONTROL:**

QUALITY CONTROL DATA FROM 08/14/02 TO 11/18/02

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| C: | 6 | 0.50 | 0.50001 | 0.00001 | 0.000008 |
| D: | 6 | 0.05 | 0.05000 | 0.00000 | 0.000005 |
| C+D: | | 0.55 | 0.55000 | 0.00000 | 0.000009 |
| C-D: | | 0.45 | 0.45001 | 0.00001 | 0.000009 |

s.d.(CD) S(between runs): 0.00001 Sw(within run): 0.00001 S/Sw: 1.00

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

0.55004 - 0.54996 for C+D
0.45004 - 0.44998 for C-D

**SOLIDS, SUSPENDED IGNITED ( PARTICULATE ASH )**

**DUPLICATES:**

| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 25 | 0 - 100.0 | 0.7366 | 8.7 |
| 0 | 100.1 - 500.0 | N.A. | N.A. |
| 0 | 500.1 - 1000.0 | N.A. | N.A. |
| 2 | 1000.1 - 5000.0 | N.A. | N.A. |
| 27 | Overall | 5.0926 | |

**OTHER CHECKS:**

| | n | Data Mean (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| Blank | 13 | -0.4792 | 0.4038 |

**SOLIDS, SUSPENDED IGNITED ( PARTICULATE LOSS ON IGNITION )**

**DUPLICATES:**

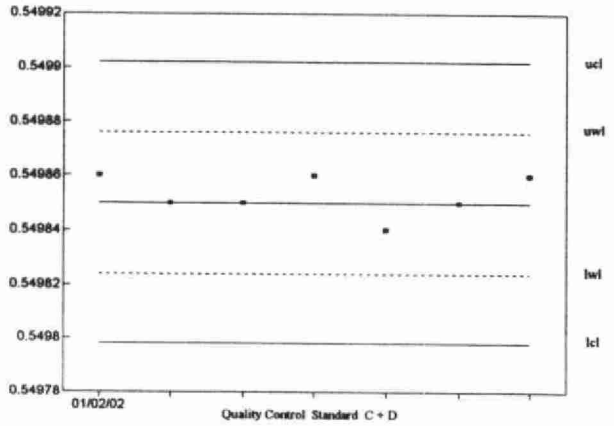
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 25 | 0 - 50.0 | 0.5019 | 7.0 |
| 0 | 50.1 - 100.0 | N.A. | N.A. |
| 0 | 100.1 - 1000.0 | N.A. | N.A. |
| 2 | 1000.1 - 5000.0 | N.A. | N.A. |
| 27 | Overall | 7.8806 | |

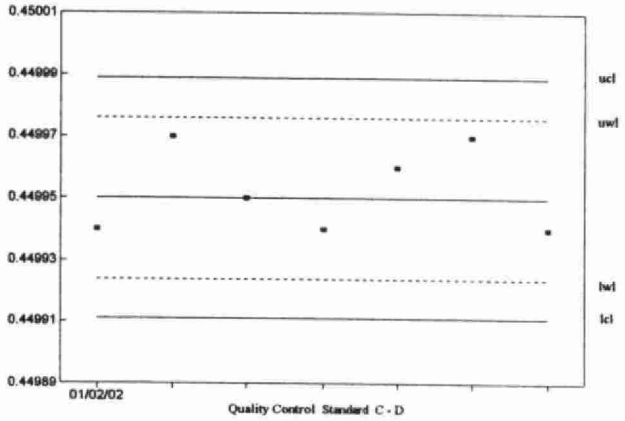
**OTHER CHECKS:**

| | n | Data Mean (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| Blank | 13 | 0.0769 | 0.1804 |

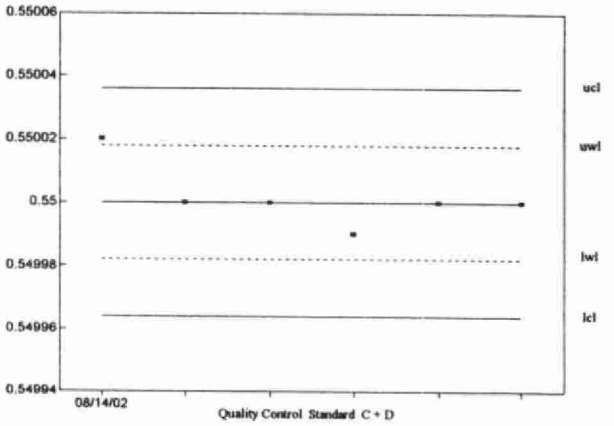
## Solids, Suspended Ignited (E3188)
(Particulate Ash and Particulate Loss on Ignition)

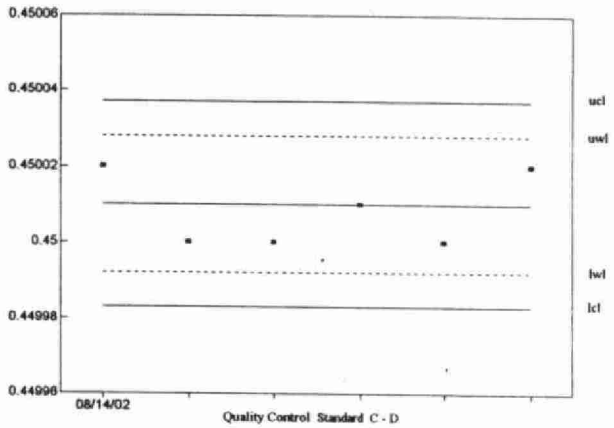
QUALITY CONTROL DATA FROM 01/02/02 TO 07/26/02




QUALITY CONTROL DATA FROM 08/14/02 TO 11/18/02



# SOLIDS, TOTAL

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '81 |
| Method Reference No. | E3188 | Reporting Unit | mg/L or mg/Kg |
| LIMS Product Code | TS3188 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 125 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Generally, 100 mL aliquot of sample (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The total residue or solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a computer system.

**INSTRUMENTATION:**

Balance (5 decimal places), drying oven, dishes (Teflon).
Computer system with appropriate software.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 2.0 | Current T value: 10 |

**CALIBRATION:**

Balance zero
Balance internal calibration performed daily.

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams) |
| Drift | Balance is reset to zero after every 10 weighings by the microcomputer. |
| Recovery | 2 standards, e.g. R1 |

## SOLIDS, TOTAL (E3188)

QUALITY CONTROL DATA FROM 01/08/02 TO 08/07/02

**CALIBRATION CONTROL:** (QC data from TS3188)

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 8 | 50.00 | 50.0006 | 0.0006 | 0.00005 |
| B: | 8 | 30.00 | 30.0004 | 0.0004 | 0.00005 |
| A+B: | | 80.00 | 80.0010 | 0.0010 | 0.00005 |
| A-B: | | 20.00 | 20.0002 | 0.0002 | 0.00008 |

s.d.(AB) S(between runs): 0.00005 Sw(within run): 0.00005 S/Sw: 0.87

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.00068 - 80.0013 for A+B
19.99996 - 20.0004 for A-B

QUALITY CONTROL DATA FROM 08/28/02 TO 12/31/02

**CALIBRATION CONTROL:** (QC data from TS3188)

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 5 | 50.00 | 50.0001 | 0.0001 | 0.00021 |
| B: | 5 | 30.00 | 30.0001 | 0.0001 | 0.00013 |
| A+B: | | 80.00 | 80.0001 | 0.0001 | 0.00033 |
| A-B: | | 20.00 | 20.0000 | 0.0000 | 0.00010 |

s.d.(AB) S(between runs): 0.00017 Sw(within run): 0.00007 S/Sw: 2.47

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

79.9997 - 80.0005 for A+B
19.9997 - 20.0003 for A-B

**RECOVERIES:**

| Number of Data | Expected Concentration (mg/L) | Mean Concentration Measured (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| 13 | 20000.0 | 20113.46 | 56.5238 |
| 13 | 2000.0 | 1996.67 | 9.9586 |

**DUPLICATES:**

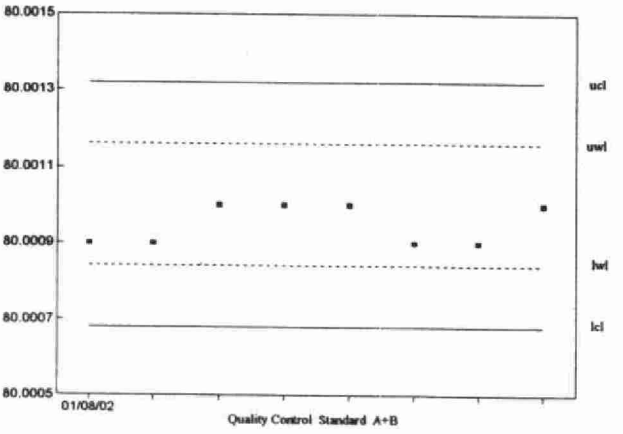
| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 11 | 0 - 6000 | 102.5481 | 5.8 |
| 8 | 6001 - 25000 | 218.7225 | 1.6 |
| 3 | 25001 - 50000 | 237.2099 | 0.8 |
| 22 | Overall | 174.1471 | |

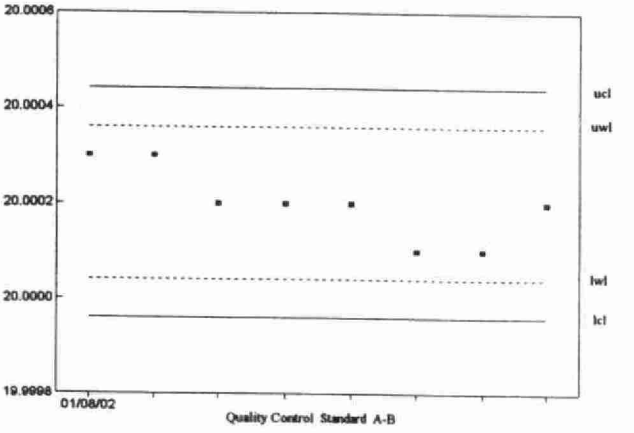
**OTHER CHECKS:**

| | n | Data Mean (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| Blank | 13 | -3.0885 | 4.1498 |

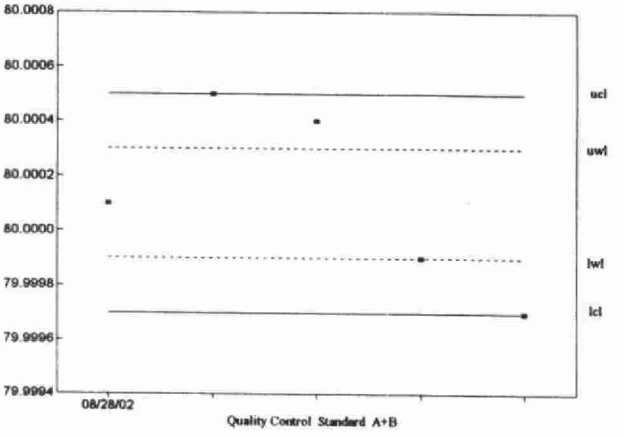
## SOLIDS, TOTAL (E3188)

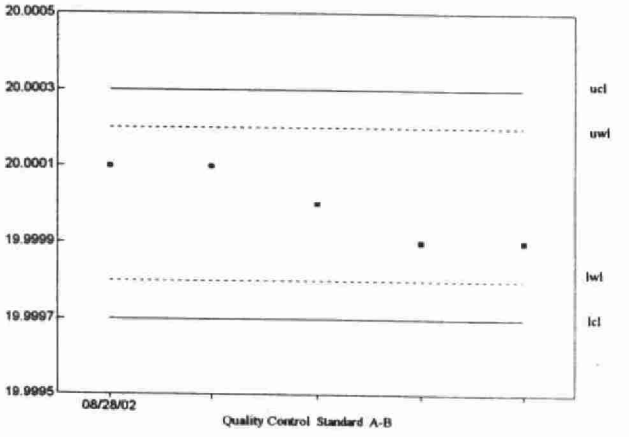
### Quality Control Data From 02/08/02 To 08/07/02




### Quality Control Data From 08/28/02 To 12/31/02



## SOLIDS, TOTAL IGNITED
## ( Ash and Loss On Ignition)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before '61 |
| Method Reference No. | E3188 | Reporting Unit | mg/L |
| LIMS Product Code | TIGN3188 | Supervisor | P. Wilson |
| Sample Type/Matrix | Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 5-500 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

The procedure for total solids (TS3188) is followed and the dried residue is ignited at 600±50°C for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for TS3188. The loss on ignition (estimate of volatile total solids) is the difference between the final ignited mass plus filter and the residue (total solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a computer system.

**INSTRUMENTATION:**

Balance (5 decimal places), muffle furnace, filters, Petri dishes.
Computer system with appropriate software.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 2.0 | Current T value: 10 |

**CONTROLS:**

| | |
|---|---|
| Calibration | 2 S class weights, e.g. QCA (results in grams) |
| Drift | Balance is reset to zero after every 10 weighings by the microcomputer. |

## SOLIDS, TOTAL IGNITED (E3188)
## (Ash and Loss On Ignition)

QUALITY CONTROL DATA FROM 01/08/02 TO 08/06/02

### CALIBRATION CONTROL:

| | n | Expected Mass (g) | Mean Mass Measured (g) | Mean Bias (g) | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 14 | 50.00 | 50.0006 | 0.0006 | 0.00004 |
| B: | 14 | 30.00 | 30.0003 | 0.0003 | 0.00005 |
| A+B: | | 80.00 | 80.0009 | 0.0009 | 0.00007 |
| A-B: | | 20.00 | 20.0002 | 0.0002 | 0.00006 |

s.d.(AB) S(between runs): 0.00005 Sw(within run): 0.00004 S/Sw: 1.05

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0007 - 80.0011 for A+B
20 - 20.0004 for A-B

### SOLIDS, TOTAL IGNITED ( DRY )

#### RECOVERIES:

| Number of Data | Expected Concentration (mg/L) | Mean Concentration Measured (mg/L) | Standard Deviation (1) |
|---|---|---|---|
| 14 | 20000.0 | 20143.3 | 142.04 |
| 14 | 2000.0 | 2004.5 | 14.28 |

#### DUPLICATES:

| n Data Pairs | Sample Concentration Span (mg/L) | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 2 | 0 - 10000 | N.A. | N.A. |
| 4 | 10001 - 15000 | 247.85 | 1.9 |
| 5 | 15001 - 25000 | 170.03 | 0.8 |
| 12 | 25001 - 50000 | 298.29 | 0.8 |
| 23 | Overall | 259.82 | |

## SOLIDS, TOTAL IGNITED cont'd
## (Ash and Loss On Ignition)

### SOLIDS, TOTAL IGNITED ( ASH )

**DUPLICATES:**

| n<br>Data Pairs | Sample<br>Concentration Span (mg/L) | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 7 | 0 - 5000 | 28.48 | 0.6 |
| 9 | 5001 - 15000 | 69.41 | 0.7 |
| 7 | 150001 - 25000 | 209.08 | 1.0 |
| 23 | Overall | 126.17 | |

**OTHER CHECKS:**

| Ashed | n | Data<br>Mean (mg/L) | Standard<br>Deviation (1) |
|---|---|---|---|
| Blank | 14 | 0.8636 | 6.4693 |

### SOLIDS, TOTAL IGNITED ( LOSS ON IGNITION )

**DUPLICATES:**

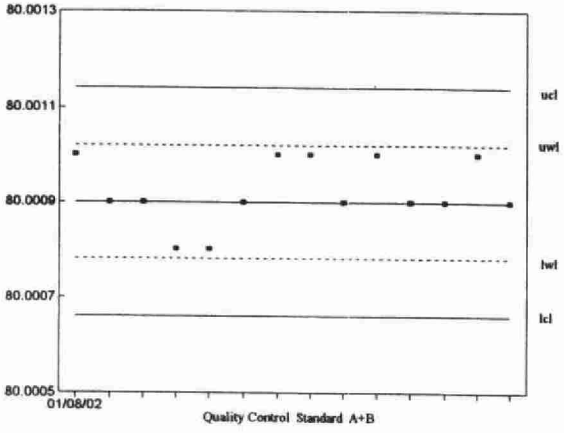
| n<br>Data Pairs | Sample<br>Concentration Span (mg/L) | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 1 | 0 - 5000 | N.A. | N.A. |
| 12 | 5001 - 15000 | 209.37 | 2.1 |
| 9 | 15001 - 25000 | 200.29 | 1.0 |
| 2 | 25001 - 50000 | N.A. | N.A. |
| 24 | Overall | 203.46 | |

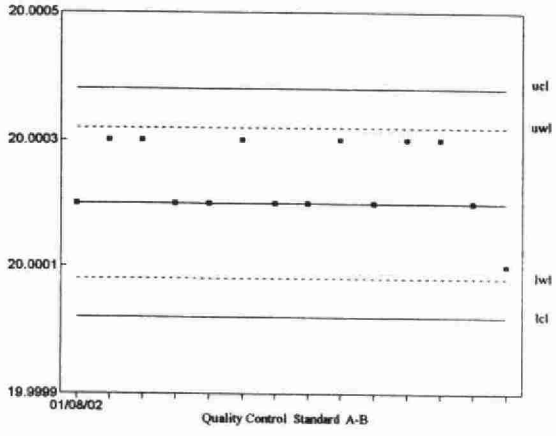
**OTHER CHECKS:**

| LOI | n | Data<br>Mean (mg/L) | Standard<br>Deviation (1) |
|---|---|---|---|
| Blank | 14 | 0.4829 | 4.0551 |

## SOLIDS, TOTAL IGNITED (E3188)

Quality Control Data From 01/08/02 To 08/06/02



# SULPHATE

## IDENTIFICATION:

| | | | |
|---|---|---|---|
| Laboratory Unit | Water Chemistry | Method Introduced | 01/04/78 |
| Method Reference No. | E3004 | Units | $\mu g/m^3$ as $SO_4$ |
| LIMS Product Code | ANION3004 | Supervisor | P. Wilson |
| Sample Type/Matrix | Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff | | |

## SAMPLING:

| | |
|---|---|
| Quantity Required | 3/4" or 1.9cm strip from 8"x10" filter |
| Container | 50 mL polypropylene tube |

## SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

## ANALYTICAL PROCEDURE:

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu g/m^3$ as $SO_4$. Chloride and nitrate are determined simultaneously.

## INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

## REPORTING:

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.1 $\mu g/m^3$ | Current T value: 0.5 $\mu g/m^3$ |

## CALIBRATION:

6 standards

## CONTROLS:

| | |
|---|---|
| Calibration | MB, IS(n), CS1, and CS2 |
| Drift | Duplicate plus 2 standards approximately every 20 samples |
| Recovery | CS3 & CS4 |

**NOTES:**

To convert unit from mg/L to $\mu g/m^3$, the final concentration of $SO_4$ in mg/L is multiplied by the following formula:

Result (mg/L) X 50mL X (63/6.75) / air volume = $\mu g/m^3$

where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

**SULFATE** (E3004)

QUALITY CONTROL DATA FOR 01/17/02 TO 12/31/02

Analytical Range: to 28.61 µg/m$^3$

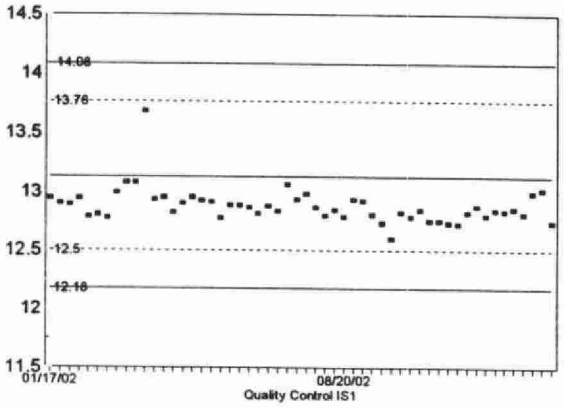
**DUPLICATES:**

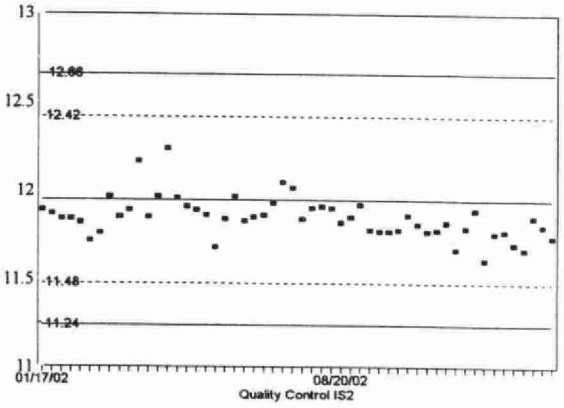
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 40 | 0.00 - 2.86 | 0.0500 | 3.2 |
| 10 | 2.89 - 7.15 | 0.0671 | 1.8 |
| 7 | 7.18 - 14.31 | 0.1832 | 1.6 |
| 13 | 14.33 - 28.61 | 0.1808 | 1.1 |
| 70 | Overall | 0.1072 | |

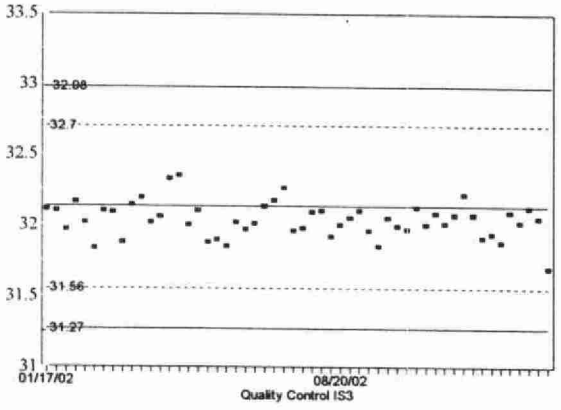
## SULPHATE (E3004)

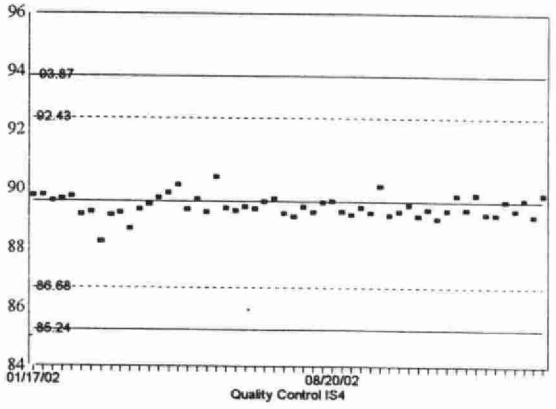
QUALITY CONTROL DATA FROM 01/17/02 TO 12/31/02

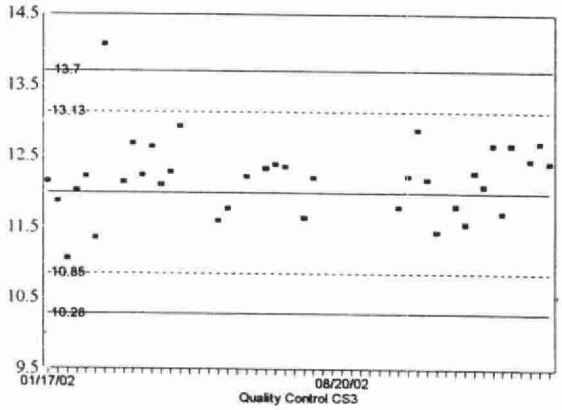
Analytical Range For IS Controls: to 100 mg/L
Analytical Range For CS Controls: to 28.61 µg/m$^3$

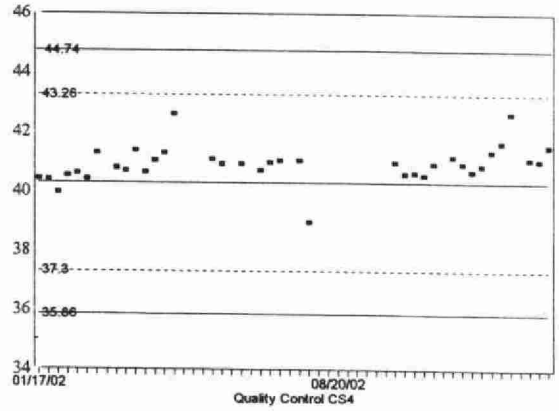


# SULPHATE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory Unit | Water Chemistry | Method Introduced | 31412 |
| Method Reference No. | E3013 | Units | µg/g as $SO_4$ |
| LIMS Product Code | ANION3013, SUL3013 | Supervisor | P. Wilson |
| Sample Type/Matrix | Soil and Sediment | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 20 g |
| Container | glass or plastic |

**SAMPLING PREPARATION:**

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

**ANALYTICAL PROCEDURE:**

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as $SO_4$.
Chloride is determined simultaneously.

**INSTRUMENTATION:**

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 µg/g | Current T value: 2.5 µg/g |

**CALIBRATION:**

8 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | MB, IS(n), CS1, and CS2 |
| Drift | Duplicate plus 2 standards approximately every 20 samples |

**SULFATE** (E3013)

QUALITY CONTROL DATA FOR 2002

Analytical Range: to 1000 µg/g

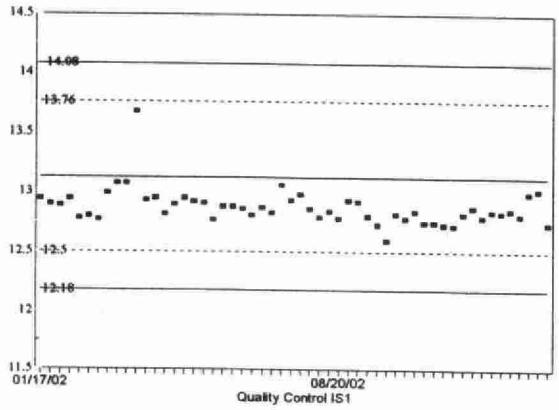
**DUPLICATES:**

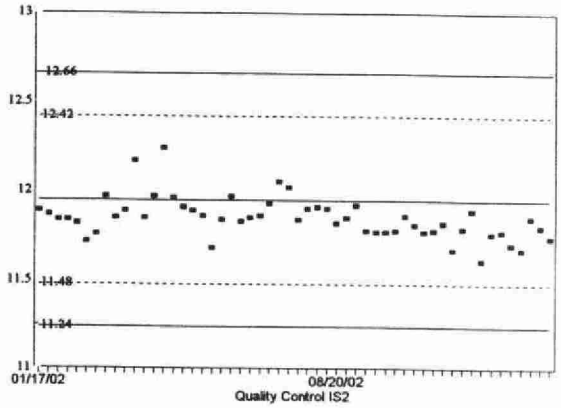
| n<br>Data Pairs | Sample<br>Concentration Span | Standard<br>Deviation (2) | Coefficient of<br>variation(%) |
|---|---|---|---|
| 12 | 0.00 - 200 | 0.4627 | 0.9 |
| 0 | 201 - 500 | N.A. | N.A. |
| 0 | 501 - 1000 | N.A. | N.A. |
| 12 | Overall | 0.4627 | |

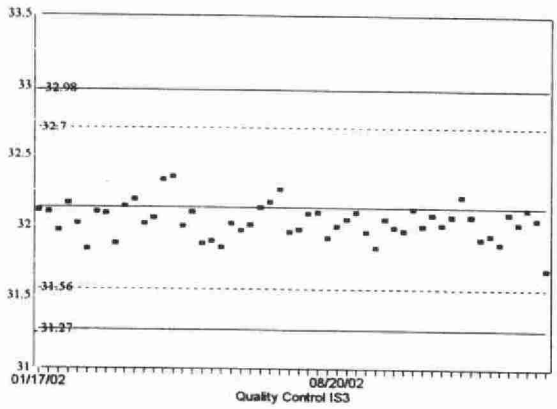
## SULPHATE (E3013)

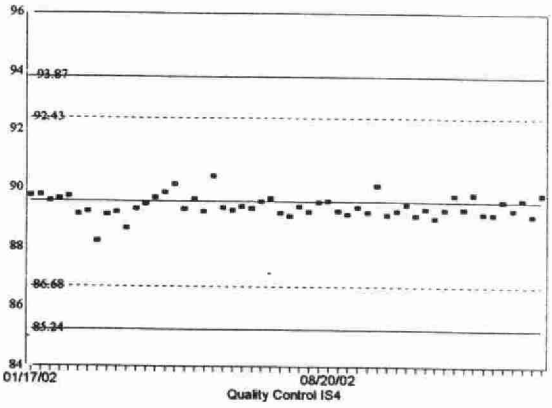
QUALITY CONTROL DATA FROM 01/17/02 TO 12/31/02

Analytical Range For IS Controls: to 100 mg/L



# SULPHATE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | 30041 |
| Method Reference No. | E3172 | Reporting Unit | mg/L as $SO_4$ |
| LIMS Product Code | SULP3172, Anion3172 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Surface Water, Ground Water, Leachates, Effluent, Industrial Waste, Raw Sewage | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**

Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.001 M sodium bicarbonate and 0.0035 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as $SO_4$ is determined by comparison of the sample scan to a series of standard scans.

**INSTRUMENTATION:**

Basic modular continuous flow ion chromatographic system, Justice Innovation ChromPerfect Spirit Data Station, plus control module (in-house design) for automated sample introduction, timing and detector range switching.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.5 | Current T value: 2.5 |

**CALIBRATION:**

BL plus 9 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | LTBL plus 3 standards, e.g. QCA |
| Drift | CHK1 and CHK2 standard approximately every 20 samples |

## SULPHATE (E3172)

QUALITY CONTROL DATA FROM 01/07/02 TO 12/27/02

Analytical Range: to 100.0 mg/L as $SO_4$

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 87 | 80.0 | 79.44 | -0.56 | 0.35 |
| B: | 87 | 40.0 | 39.94 | -0.06 | 0.23 |
| C: | 87 | 8.0 | 7.86 | -0.14 | 0.15 |
| A+B: | | 120.0 | 119.38 | -0.62 | 0.44 |
| A-B: | | 40.0 | 39.51 | -0.49 | 0.39 |
| B+C: | | 48.0 | 47.79 | -0.21 | 0.32 |
| B-C: | | 32.0 | 32.08 | 0.08 | 0.23 |

| | | | | |
|---|---|---|---|---|
| s.d.(AB) | S(between runs): 0.30 | Sw(within run): | 0.28 | S/Sw: 1.07 |
| s.d.(BC) | S(between runs): 0.19 | Sw(within run): | 0.16 | S/Sw: 1.20 |

The calibration is accepted if the calibration control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 117.3 | - | 122.7 | for | A+B |
| 37.97 | - | 42.03 | for | A-B |
| 46.89 | - | 49.11 | for | B+C |
| 31.17 | - | 32.83 | for | B-C |

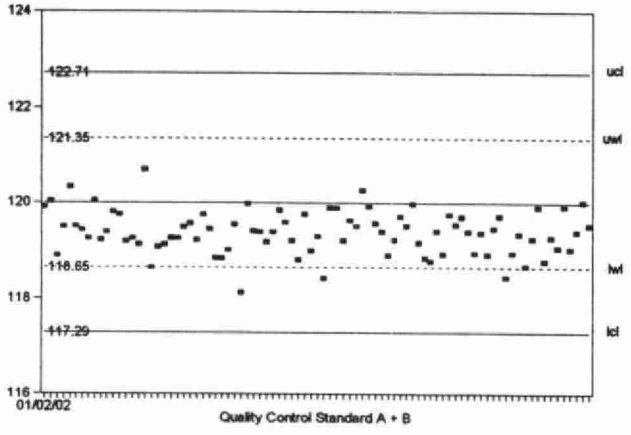
**DUPLICATES:**

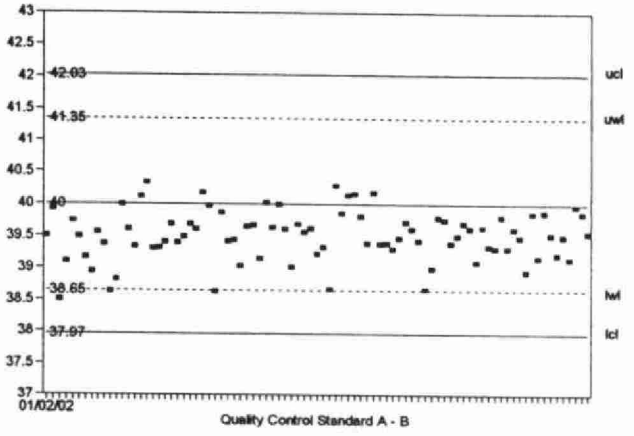
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 42 | 0.0 - 10.0 | 0.1393 | 3.0 |
| 54 | 10.1 - 20.0 | 0.2552 | 1.7 |
| 92 | 20.1 - 50.0 | 0.3787 | 1.2 |
| 27 | 50.1 - 100.0 | 0.5392 | 0.8 |
| 215 | Overall | 0.3436 | |

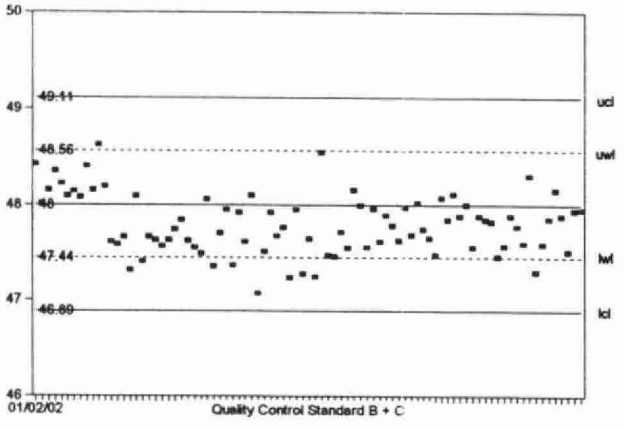
## SULPHATE (E3172)

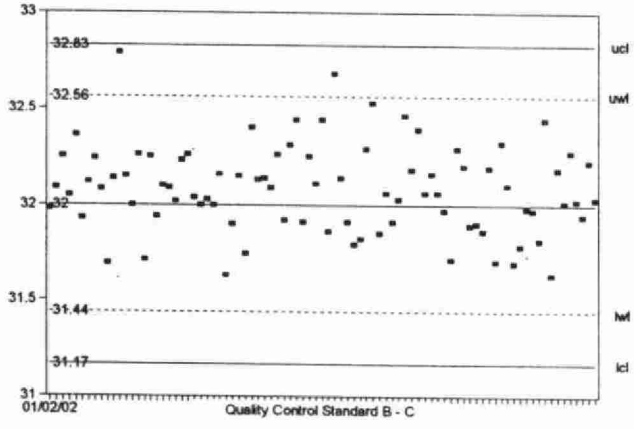
QUALITY CONTROL DATA FROM 01/07/02 TO 12/27/02

Analytical Range: to 100.0 mg/L as $SO_4$



## SULPHIDE

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | June 89 |
| Method Reference No. | E3100 | Reporting Unit | µg/L as $S^{2-}$ |
| LIMS Product Code | H2S3100 | Supervisor | P.Wilson |
| Sample Type/Matrix | Drinking Water, Surface Water, Ground Water, Leachates, Effluent, Industrial Waste, Raw Sewage | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required | 50 mL |
| Container | Glass or plastic |

**ANALYTICAL PROCEDURE:**
Total Sulphide ($H_2S$, $HS^-$ and any acid soluble metal sulphides) have been precipitated as ZnS during sample preservation. The precipitated sulphides (hydrogen sulphides) are dissolved in an alkaline absorbing solution and reacted with N,N-dimethyl-p-phenylenediamine dihydrochloride and ferric chloride to form methylene blue. The intensity of the methylene blue is compared to standards treated in the same manner.

**INSTRUMENTATION:**
Basic automated modular continuous flow colourimetric system, measurement through a 660 nm filter and a 50 mm flow cell (1.5mm ID).

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 2.0 µg/L | Current T value: 10.0 µg/L |

**CALIBRATION:**
BL plus 5 standards

**CONTROLS:**

| | |
|---|---|
| Calibration | Daily blank and 3 standards, e.g. QCA |
| Drift | Sensitivity check standard approximately every 10 samples |

## SULPHIDE (E3100)

QUALITY CONTROL DATA FROM 02/28/01 TO 12/05/02

Analytical Range: to 100.0 µg/L as $S^{2-}$

**CALIBRATION CONTROL:**

| | n | Expected Concentration | Mean Concentration | Mean Bias | Standard Deviation (1) |
|---|---|---|---|---|---|
| A: | 18 | 128 | 125.37 | -2.63 | 13.04 |
| B: | 18 | 80 | 80.87 | 0.87 | 10.24 |
| C: | 18 | 32 | 35.46 | 3.46 | 10.54 |
| A+B: | | 208 | 206.24 | -1.76 | 22.29 |
| A-B: | | 48 | 44.50 | -3.50 | 7.29 |
| B+C: | | 112 | 116.32 | 4.32 | 16.87 |
| B-C: | | 48 | 45.41 | -2.59 | 12.12 |

| | | | | |
|---|---|---|---|---|
| s.d.(AB) | S(between runs): 11.72 | Sw(within run): | 5.16 | S/Sw: 2.3 |
| s.d.(BC) | S(between runs): 10.39 | Sw(within run): | 8.57 | S/Sw: 1.2 |

The calibration is accepted if the calibration control values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 179.2 | - | 236.8 | for | A+B |
| 26.4 | - | 69.6 | for | A-B |
| 62.3 | - | 161.7 | for | B+C |
| 10.7 | - | 85.3 | for | B-C |

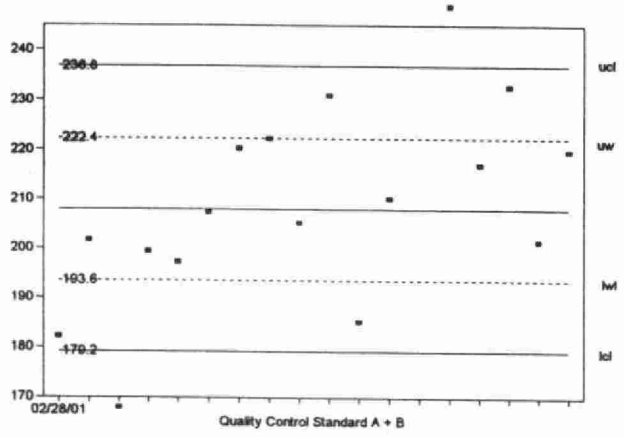
**DUPLICATES:**

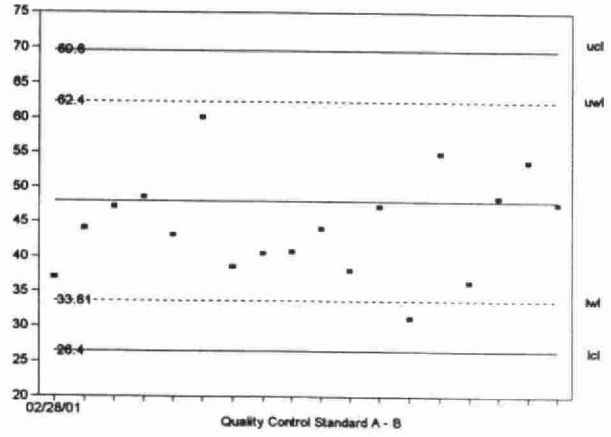
| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 14 | 0.0 - 32.0 | 0.8194 | 6.1 |
| 7 | 32.1 - 80.0 | 3.3438 | 6.0 |
| 17 | 80.1 - 160.0 | 1.5253 | 1.4 |
| 38 | Overall | 1.8297 | |

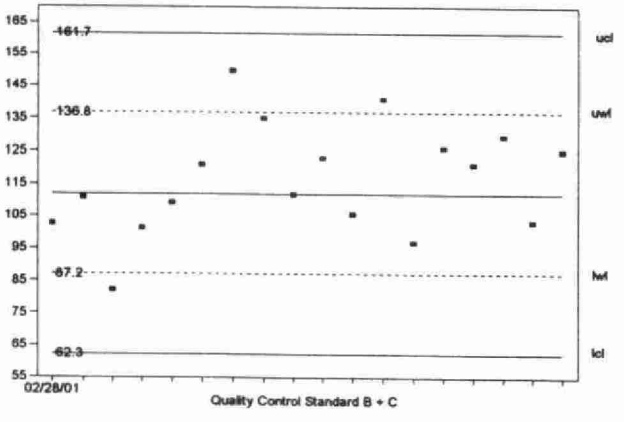
## SULPHIDE (E3100)

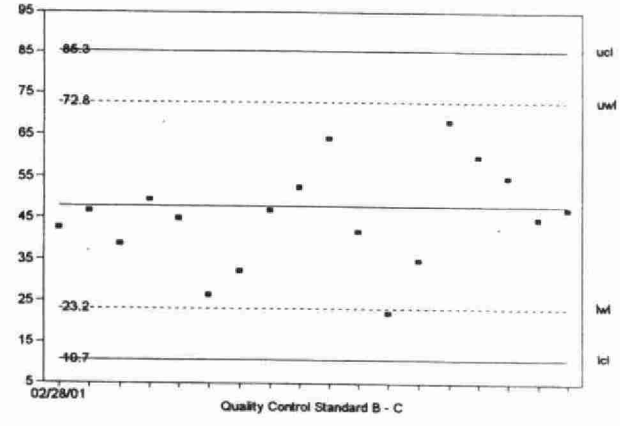
QUALITY CONTROL DATA FROM 02/28/01 TO 12/05/02

Analytical Range: to 100.0 µg/L as $S^{2-}$




Note:

For explanation of any exceedence, refer to raw data file.

# TURBIDITY

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Water Chemistry | Method Introduced | Before'74 |
| Method Reference No. | E3311 | Reporting Unit | FTU |
| LIMS Product Code | TURB3311 | Supervisor | P. Wilson |
| Sample Type/Matrix | Surface Water, Ground Water, Effluent, Drinking Water, Industrial Waste, Process Water, Leachate | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 50 mL |
| Container: | Glass or plastic |

**ANALYTICAL PROCEDURE:**

The instrument is standardized with sealed standards which are prepared commercially and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurements are based on light scattering at 90° ( ±30°) rotation. The instrument compensates for sample colour.

**INSTRUMENTATION:**

-Hach Ratio/XR Model Turbidimeter modified to accept control signals from robot controller, electronic interface, Zymark ZYMATE 11 Laboratory Robot System and computer.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 3 | Current W value: 0.05 | Current T value: 0.25 |

**CALIBRATION:**

BL plus formazin standards (once every four months)

**CONTROLS:**

| | |
|---|---|
| Calibration: | 5 standards, e.g. QCA |

**NOTES:** QCD data for September 18th , October 3rd and 4th are outside the limits. Repeat analysis was done on the samples in that range for September 18th. There are no samples in that range for October 3rd and 4th .

## TURBIDITY (E3311)

QUALITY CONTROL DATA FROM 01/02/02 TO 12/24/02

Analytical Range: to 2000 FTU

**CALIBRATION CONTROL:**

January to October:

| | n | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| A: | 184 | 2.0 | 1.4558 | 0.1820 |
| B: | 184 | 20.0 | 15.5446 | 0.2623 |
| C: | 146 | 200.0 | 150.0182 | 1.6814 |
| D: | 146 | 2000.0 | 1337.699 | 9.6921 |

October to December:

| | n | Expected Concentration | Mean Concentration | Standard Deviation (1) |
|---|---|---|---|---|
| C: | 38 | 200.0 | 184.3763 | 1.0916 |
| D: | 38 | 2000.0 | 1584.237 | 6.4449 |

On any given day the calibration is accepted if the values obtained lie within the ranges:

| | | | | |
|---|---|---|---|---|
| 1.3582 | - | 1.8487 | for | A (Jan-Apr) |
| 1.4727 | - | 1.5880 | | A (May-Aug) |
| 1.5012 | - | 1.5817 | | A (Sep-Nov) |
| 1.0679 | - | 1.1332 | | A (Dec) |
| 15.152 | - | 15.974 | for | B (Jan-Apr) |
| 14.7846 | - | 15.5683 | | B (May-Aug) |
| 14.7408 | - | 16.0025 | | B (Sep-Nov) |
| 15.4371 | - | 16.1296 | | B (Dec) |
| 149.46 | - | 153.79 | for | C (Jan-Apr) |
| 145.4449 | - | 151.7615 | | C (May-Aug) |
| 145.3628 | - | 154.4239 | | C (Sep-Nov) |
| 180.6761 | - | 188.3706 | | C (Dec) |
| 1330.1 | - | 1373.5 | for | D (Jan-Apr) |
| 1302.181 | - | 1365.174 | | D (May-Aug) |
| 1329.537 | - | 1348.663 | | D (Sep-Nov) |
| 1564.563 | - | 1602.37 | | D (Dec) |

| | n | Data Mean | Standard Deviation (1) |
|---|---|---|---|
| Stray Light | 184 | 0.015 | 0.0039 |

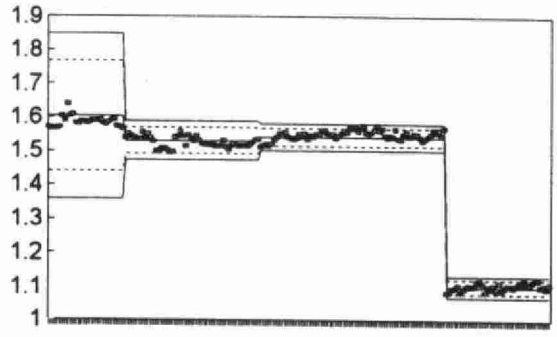
**DUPLICATES:**

| n Data Pairs | Sample Concentration Span | Standard Deviation (2) | Coefficient of variation(%) |
|---|---|---|---|
| 326 | 0.0 - 2.0 | 0.0446 | 7.5 |
| 176 | 2.1 - 20.0 | 0.2993 | 4.6 |
| 41 | 21.0 - 200 | 1.3751 | 2.5 |
| 2 | 201 - 2000 | N.A. | N.A. |
| 544 | Overall | 0.5598 | |

## TURBIDITY (E3311)

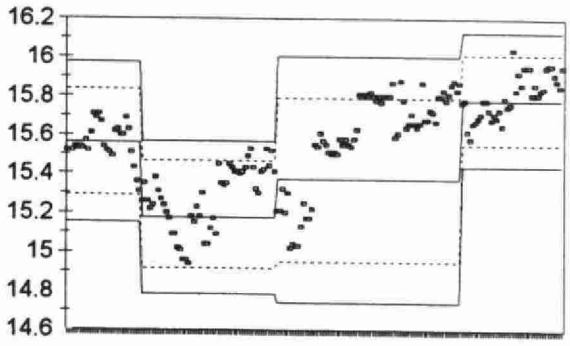
QUALITY CONTROL DATA FROM 01/02/02 TO 12/24/02

Analytical Range: to 2000 FTU



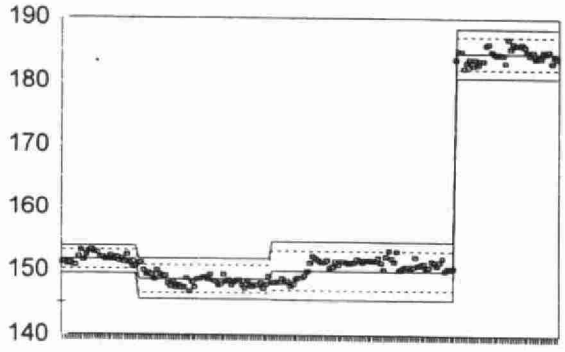

Quality Control Standard A

Jan-Apr May-Aug Sept - Nov Dec



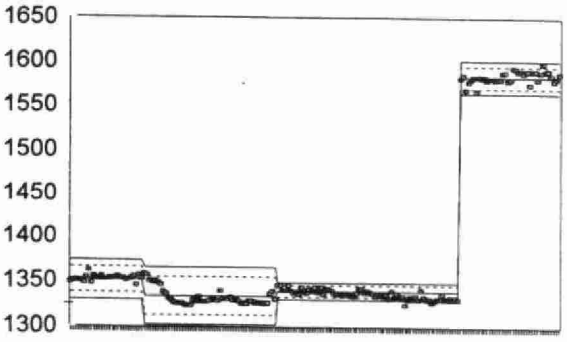

Quality Control Standard B

Jan-Apr May-Aug Sept - Nov Dec




Quality Control Standard C

Jan-Apr May - Aug Sept - Nov Dec




Quality Control Standard D

Jan-Apr May -Aug Sept - Nov Dec

# PART 3.0

# MICROBIOLOGY

## 3.1 Quality Control Program, Microbiology Unit

### Performance Criteria

Analyses of samples in the Microbiology Unit are performed using approved methodologies, by trained technologists. Safety measures have been incorporated into the methodologies to ensure that all analytical procedures are functioning properly, minimizing the potential to identify and report false positive or negative results. This report focuses on the quality control implemented during sample analyses. Information regarding the implementation of quality control procedures for sample containers, monitoring of the Pure Water supply, media preparation and storage, equipment monitoring are described by the Laboratory Services Branch (4) and Microbiology Unit Standard Operating Procedures (SOPs), approved Microbiology Methods and Lab Services Branch Quality Assurance Manual (2).

### Membrane Filtration

Blank Control Analyses

A control (sterile buffered dilution water) sample is processed between each sample analyzed. The control sample is processed in a manner similar to the regular sample including volume, agar used, incubation time and temperature. The blank control should remain free of any bacterial growth.

Duplicate Analyses

Approximately five percent of the samples are analyzed in duplicate per day. The data are accumulated for each parameter and a "within-run" standard deviation is calculated to give a measure of the repeatability of the results.

### Presence-Absence Procedure

Blank Control Analyses

Approximately five percent of samples analyzed per day include, a blank control sample prepared by adding a 99 mL dilution blank (sterile, buffered dilution water) to P-A broth and incubating it along with the regular P-A bottles. The blank control should remain free of any bacterial growth and there should be no change in the colour of the broth. Identification of growth or colour change in the control blank requires follow-up of sterility checks in both the P-A broth and the dilution blanks.

### Heterotrophic Spread Plate

Blank Control Analyses

Approximately five percent of samples analyzed per day include inoculating a Plate Count agar plate with 0.1 mL of sterile buffered dilution water and incubating it along with the regular Plate Count agar plates (35±0.5°C, 48±3 hours).

Duplicate Analyses

Approximately five percent of samples are analyzed in duplicate per day. The data are accumulated for each parameter and a "within-run" standard deviation is calculated to give a measure of the repeatability of the results.

Blank Analyses Corrective Action

The presence of bacterial growth on any control sample by the above techniques (Membrane Filtration, PA Broth, Heterotrophic Spread Plate) indicates inaccurate technique. The supervisor must be consulted with regards to determining follow-up and corrective action. Reporting of results may be tempered by the presence of bacterial growth on these control samples and data qualifying remarks codes would be noted on the final report. Records of all control samples are maintained in the laboratory.

## 3.2 PERFORMANCE SUMMARIES

## MICROBIOLOGY

## *Escherichia coli* (EC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3226 | Reporting Unit | Present/Absent per 100 mL |
| LIMS Product Code | PA3226 | Supervisor | R. Schop |
| Sample Type/Matrix | Drinking Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are added to P-A broth, incubated (35±0.5°C, for up to 72±3 hours ) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for *Escherichia coli* are conducted according to the method.

**INSTRUMENTATION:**

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators, UV lamp

**REPORTING:**

| |
|---|
| Present / Absent per 100 mL |

**CONTROLS:**

| | |
|---|---|
| Analytical | Negative Control(5% per day) -Sterile buffered dilution water |

**NOTES:**

*PA3226 is used for the detection of EC and TC. Confirmatory testing using ECMug is required.

*Escherichia coli* (EC)
**E3226**

QUALITY CONTROL DATA FOR 2002

Present/Absent per 100 mL

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 190 | 0 |

## *Escherichia coli* (EC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3371 | Reporting Unit | CFU per 100 mL |
| LIMS Product Code | EC3371, *TCEC3371,*ECFS3371*, ECFSPS3371 | Supervisor | R. Schop |
| Sample Type/Matrix | Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto MFC-BCIG agar plate and incubated 44.5±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

* TCEC3371,*ECFS3371*,ECFSPS3371 are mixed parameter product codes. See individual tests TC,FS,PSA, for details on medium used and incubation.

*Escherichia coli* (EC)
E3371

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| n Data Pairs | Counts per Plate | Mean Difference | Standard Deviation (2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 67 | 0-30* | 2.12 | 1.98 | 22.69 |
| 14 | 31-75 | 3.92 | 3.58 | 7.74 |
| 6 | 76-150 | 6.83 | 6.55 | 6.23 |

* 27 duplicates pairs with counts per filter of zero on each, were not included in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 1622 | 0 |

## *Escherichia coli* (EC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1998 |
| Method Reference No. | E3407 | Reporting Unit | CFU per 100mL |
| LIMS Product Code | *TCEC3407 | Supervisor | R. Schop |
| Sample Type/Matrix | Drinking Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto DC agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, bunsen burner, incubator, microscope, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

*TCEC3407 is a mixed parameter product code. The same medium and incubation time are used to determine both parameters TC and EC.

*Escherichia coli* (EC)
**E3407**

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| n Data Pairs | Counts per Plate | Mean Difference | Standard Deviation (2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 3 | 0-30* | 1.33 | 1.00 | 23.08 |
| 3 | 31-75 | 6.33 | 5.58 | 14.63 |
| 0 | 76-150 | N.A. | N.A. | N.A. |

* 16 duplicates pairs with counts per filter of zero on each, were not included in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 235 | 0 |

# FAECAL STREPTOCOCCI (FS)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3371 | Reporting Unit | CFU per 100 mL |
| LIMS Product Code | FS3371,*ECFS3371, *ECFSPS3371 | Supervisor | R. Schop |
| Sample Type/Matrix | Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mEnterococcus agar plate and incubated 35±0.5°C, 48±3 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

*ECFS3371*,ECFSPS3371 are mixed parameter product codes. See individual tests EC,PSA, for details on medium used and incubation.

**FAECAL STREPTOCCOCI (FS)**
**E3371**

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| (n) Data Pairs | Counts per Plate | Mean Difference | Standard Deviation (2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 54 | 0-30* | 2.67 | 2.49 | 30.52 |
| 14 | 31-75 | 3.57 | 3.07 | 7.40 |
| 7 | 76-150 | 6.43 | 5.44 | 8.22 |

* 8 duplicates pairs with counts per filter of zero on each, were not included in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 1178 | 0 |

# HETEROTROPHIC PLATE COUNT(HPC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1998 |
| Method Reference No. | E3408 | Reporting Unit | CFU per 1mL |
| LIMS Product Code | PC3408 | Supervisor | R. Schop |
| Sample Type/Matrix | Drinking Water, Ground Water, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquot is inoculated onto a Plate Count agar plate with a micropipette. The sample is then spread onto the plate using a glass rod and an electronic turntable. The plate is then incubated 35±0.5°C, 48±3 hours and checked for growth. Target colonies formed on the plate are recorded per 1 mL of sample

**INSTRUMENTATION:**

Micropipette, sterile micropipette tips, sterile glass rod, electronic turntable, incubator, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicates (5% per day)<br>Negative control per run- open air plate<br>Negative control (5% per day) - glass rod check |

## HETEROTROPHIC PLATE COUNT (HPC)
## E3408

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/1 mL

**DUPLICATES:**

| n<br>Data Pairs | Counts per Plate | Mean Difference | Standard Deviation<br>(2) | Coefficient of<br>Variation (%) |
|---|---|---|---|---|
| 79 | 0 - 30* | 1.72 | 1.52 | 30.59 |
| 3 | 31 - 75 | 3.33 | 2.77 | 19.32 |
| 1 | 76 -150 | 10.00 | N.A. | N.A. |

*106 duplicate pairs with counts per plate of zero on each were not included in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 260 | 0 |

# INDICATOR ORGANISMS

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3226 | Reporting Unit | Present/Absent per 100 mL |
| LIMS Product Code | PA3226 | Supervisor | R. Schop |
| Sample Type/Matrix | Drinking Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are added to P-A broth and incubated (35±0.5°C, for up to 72±3 hours ) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for indicator organisms are conducted according to the method.

**INSTRUMENTATION:**

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators

**REPORTING:**

| |
|---|
| Detected/Not Detected per 100 mL |

**CONTROLS:**

| | |
|---|---|
| Analytical | Negative Control(5% per day) -Sterile buffered dilution water |

**NOTES:**

*PA3226 is used for the detection of indicator organisms. Various media are used in their determinations . See method.

## *Pseudomonas aeruginosa* (PSA)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3371 | Reporting Unit | CFU per 100 mL |
| LIMS Product Code | PSA3371,*ECFSPS3371 | Supervisor | R. Schop |
| Sample Type/Matrix | Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mPA agar plate and incubated 41.5±0.5°C, 48±3 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

*ECFSPS3371 is a mixed parameter product code. See individual test EC, FS for details on medium used and incubation.

**Pseudomonas aeruginosa (PSA)**
**E3371**

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| Data Pairs (n) | Counts per Plate | Mean Difference | Standard Deviation (2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 53 | 0-30* | 2.21 | 2.14 | 27.61 |
| 3 | 31-75 | 3.00 | 2.35 | 4.08 |
| 0 | 76-150 | N.A. | N.A. | N.A. |

* 47 duplicates pairs with counts per filter of zero on each, were not included in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 1120 | 0 |

# TOTAL COLIFORM (TC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3226 | Reporting Unit | Present/Absent per 100 mL |
| LIMS Product Code | PA3226 | Scientist | R. Schop |
| Sample Type/Matrix | Drinking Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are added to P-A broth and incubated (35±0.5°C, for up to 72±3 hours ) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for Total Coliforms are conducted according to the method.

**INSTRUMENTATION:**

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators, UV lamp

**REPORTING:**

| |
|---|
| Present / Absent per 100 mL |

**CONTROLS:**

| | |
|---|---|
| Analytical | Negative Control(5% per day) -Sterile buffered dilution water |

**NOTES:**

*PA3226 is used for the detection of EC and TC. Confirmatory testing using ECMug is required.

**TOTAL COLIFORM (TC)**
**E3226**

QUALITY CONTROL DATA FOR 2002

Present/Absent per 100 mL

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 190 | 0 |

# TOTAL COLIFORM (TC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1979 |
| Method Reference No. | E3371 | Reporting Unit | CFU per 100 mL |
| LIMS Product Code | TC3371, *TCEC3371 | Supervisor | R. Schop |
| Sample Type/Matrix | Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mEndo LES agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter.

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

* TCEC3371 is a mixed parameter product code. See individual test (EC) for details on medium used and incubation.

## TOTAL COLIFORM COUNT (TC)
## E3371

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| Data Pairs | Counts per Plate | Average of all Data Points | Standard Deviation of the Duplicates | Coefficient of Variation (%) |
|---|---|---|---|---|
| 0 | 0-30 | N.A. | N.A. | N.A. |
| 0 | 31-75 | N.A. | N.A. | N.A. |
| 0 | 76-150 | N.A. | N.A. | N.A. |

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 252 | 0 |

# TOTAL COLIFORM (TC)

**IDENTIFICATION:**

| | | | |
|---|---|---|---|
| Laboratory | Microbiology | Method Introduced | 1998 |
| Method Reference No. | E3407 | Reporting Unit | CFU per 100mL |
| LIMS Product Code | *TCEC3407 | Supervisor | R. Schop |
| Sample Type/Matrix | Drinking Water | | |

**SAMPLING:**

| | |
|---|---|
| Quantity Required: | 100 mL |
| Container: | Plastic, ring sealed |
| Preservative: | Sodium thiosulphate |

**ANALYTICAL PROCEDURE:**

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto DC agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

**INSTRUMENTATION:**

Filtration assembly, sterile membrane filters, sterile graduated cylinders, bunsen burner, incubator, microscope, Quebec colony counter

**REPORTING:**

| | | |
|---|---|---|
| Maximum Significant Figures: 2 | Current W value: 0 | Current T value: Not Applicable |

**CONTROLS:**

| | |
|---|---|
| Analytical | Duplicate samples (5% per day)<br>Blank filter between samples |

**NOTES:**

*TCEC3407 is a mixed parameter product code. The same medium and incubation time are used to determine both parameters TC and EC.

**TOTAL COLIFORM (TC)**
**E3407**

QUALITY CONTROL DATA FOR 2002

Colony Forming Units/100 mL

**DUPLICATES:**

| (n) Data Pairs | Counts per Filter | Mean Difference | Standard Deviation (2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 5 | 0-30* | 4.0 | 3.32 | 47.4 |
| 5 | 31-75 | 4.2 | 4.32 | 10.6 |
| 0 | 76-150 | N.A. | N.A. | N.A. |

* 12 duplicates pairs with counts per filter pf zero on each, were not in the statistics

**OTHER CHECKS:**

| | n | number of blanks with growth |
|---|---|---|
| Control Blanks | 235 | 0 |

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## ABBREVIATIONS

| | |
|---|---|
| AAII | - Auto Analyzer Model II |
| AAS | - Atomic Absorption Spectrophotometer |
| Bl | - Blank |
| °C | - Degree Centigrade |
| cm | - Centimetre |
| CS1 | - Check Sample 1 |
| CS2 | - Check Sample 2 |
| Date | - Day/Month/Year |
| DO | - Dissolved Oxygen |
| EDTA | - Ethylenediaminetetra-Acetic Acid, Disodium Salt, Dihydrate |
| FTU | - Formazin Turbidity Units |
| g | - Gram |
| HZU | - Hazen Units |
| $in^2$ | - Square Inches |
| IS(n) | - Internal Standard (n denotes parameter) |
| kg | - kilogram |
| L | - Litre |
| LAB | - Laboratory |
| LIMS | - Laboratory Information Management System |
| LTB/L | - Long Term Blank |
| lcl | - Low Control Limit |
| lwl | - Low Warning Limit |
| $m^3$ | - Cubic Metre |
| M | - Molarity |
| MB | - Method Blank |
| meq | - Milliequivalent |
| mg | - Milligram |
| min | - Minute |
| mL | - Millilitre |
| mm | - Millimetre |
| N | - Normality |
| N.A. | - Not Available or Not Applicable |
| nm | - Nanometre |
| n | - Number |
| PC | - Personal Computer |
| Pure-DW | - Pure Deionized Water |

| | |
|---|---|
| Pure-W | - Pure Water |
| QC | - Quality Control |
| QCA | - Quality Control Standard A |
| QCB | - Quality Control Standard B |
| QCC | - Quality Control Standard C |
| QCD | - Quality Control Standard D |
| R | - Recovery |
| rpm | - Revolutions Per Minute |
| RS92 | - Reference Standard (in -house) |
| S | - Between Run Standard Deviation |
| $S_1$ | - Standard Deviation (Conventional) |
| $S_2$ | - Standard Deviation For Duplicates |
| $S_w$ | - Standard Deviation Within Run |
| S. Class | - Weight Classification Designation (not certified) |
| s.d. | - Standard Deviation |
| Standard Cal | - Colourimeter setting to control electronic expansion |
| STD | - Standard |
| TCU | - True Colour Units |
| TPTZ | - Ferrous-2,4,6-tri(2'pyridyl)-1,3,5,- triazine |
| ucl | - Upper Control Limit |
| uwl | - Upper Warning Limit |
| µm | - Micrometer |
| µeq | - Microequivalent |
| µg | - Microgram |
| µS | - Micro-Siemen |
| UV | - Ultra-Violet |
| V/V | - Concentration based on volume measurements |
| W40 | - Whatman 40 Filters |
| % | - Percent |

## Appendix A
## W and T values for '02

| Parameter | Method Reference No. | Units | Full Scale | W | T |
|---|---|---|---|---|---|
| **Alkalinity, Total Fixed Endpoint** | (E3218) | mg/L $CaCO_3$ | 1000 | 0.5 | 2.5 |
| **Carbon, Dissolved Inorganic** | (E3370) | mg/L C | 80.0 | 0.2 | 1.0 |
| **Carbon, Dissolved Organic** | (E3370) | mg/L C | 20.0 | 0.1 | 0.5 |
| **Chloride** | (E3004) | $\mu g/m^3$ Cl | 28.6 | 0.1 | 0.5 |
| **Chloride** | (E3013) | µg/g Cl | - | 0.5 | 2.5 |
| **Chloride** | (E3016) | mg/L Cl | 100 | 0.2 | 1.0 |
| **Chlorophyll "a"** | (E3169) | µg/L | - | 0.2 | 1.0 |
| **Chlorophyll "a" Acidified** | (E3169) | µg/L | - | 1.0 | 5.0 |
| **Chlorophyll "b"** | (E3169) | µg/L | - | 0.1 | 0.5 |
| **Colour, True** | (E3219) | TCU | 100 | 0.2 | 1.0 |
| **Conductivity** | (E3218) | µS/cm | 2000 | 1 | 5 |
| **Cyanide, Free** | (E3015) | mg/L $CN^-$ | 0.2 | 0.001 | 0.005 |
| | | µg/g $CN^-$ | | 0.01 | 0.05 |
| **Cyanide, Total** | (E3015) | mg/L $CN^-$ | 0.2 | 0.001 | 0.005 |
| **Cyanide, Total** | (E3015) | µg/g $CN^-$ | | 0.01 | 0.05 |
| **Fluoride** | (E3172) | mg/L F | 2.0 | 0.01 | 0.05 |
| **Nitrate** | (E3004) | $\mu g/m^3$ $NO_3$ | 28.6 | 0.1 | 0.5 |
| **Nitrilotriacetic Acid** | (E3406) | mg/L NTA | 1.00 | 0.01 | 0.05 |
| **Nitrogen,** | | | | | |
| **Ammonia Plus Ammonium** | (E3364) | mg/L N | 2.0 | 0.002 | 0.01 |
| **Ammonia Plus Ammonium** | (E3366) | mg/L N | 50.0 | 0.05 | 0.25 |
| **Nitrogen, Nitrate Plus Nitrite** | (E3364) | mg/L N | 5.00 | 0.005 | 0.025 |
| **Nitrogen, Nitrate Plus Nitrite** | (E3366) | mg/L N | 50.0 | 0.05 | 0.25 |
| **Nitrogen, Nitrite** | (E3364) | mg/L N | 0.200 | 0.001 | 0.005 |
| **Nitrogen, Nitrite** | (E3366) | mg/L N | 2.00 | 0.005 | 0.025 |
| **Nitrogen, Total Kjeldahl** | (E3116) | mg/g N | 20 | 0.1 | 0.5 |
| **Nitrogen, Total Kjeldahl** | (E3118) | mg/g N | 100 | 0.20 | 1.00 |
| **Nitrogen, Total Kjeldahl** | (E3367) | mg/L N | 2.00 | 0.02 | 0.10 |

## Appendix A
## W and T values for '02

| Parameter | Method Reference No. | Units | Full Scale | W | T |
|---|---|---|---|---|---|
| Nitrogen, Total Kjeldahl | (E3368) | mg/L N | 50.0 | 0.05 | 0.25 |
| Oxygen Demand, Biochemical | (E3182) | mg/L O | 9.0 | 0.2 | 1 |
| Oxygen Demand, Chemical | (E3170) | mg/L O | 50 | 1 | 5 |
| Oxygen Demand, Chemical | (E3246) | mg/L O | 400 | 2 | 10 |
| pH | (E3218) | - | - | - | - |
| Phenolics, Reactive | (E3179) | µg/L Phenol | 50.0 | 0.2 | 1.0 |
| Phosphorus, | | | | | |
| Reactive ortho-Phosphate | (E3364) | mg/L P | 0.100 | 0.0005 | 0.0025 |
| Reactive ortho-Phosphate | (E3366) | mg/L P | 10.0 | 0.02 | 0.10 |
| Phosphorus, Total | (E3116) | mg/g P | 2 | 0.02 | 0.10 |
| Phosphorus, Total | (E3118) | mg/g P | 25 | 0.02 | 0.10 |
| Phosphorus, Total | (E3367) | mg/L P | 0.200 | 0.002 | 0.01 |
| Phosphorus, Total | (E3368) | mg/L P | 10.0 | 0.02 | 0.10 |
| Silicon, Reactive Silicates | (E3370) | mg/L Si | 10.0 | 0.02 | 0.10 |
| Solids, Dissolved | (E3188) | mg/L | - | 2 | 10 |
| Solids, Suspended | (E3188) | mg/L | - | 0.5 | 2.5 |
| Solids, Suspended Ignited | (E3188) | mg/L | - | 0.5 | 2.5 |
| Solids, Total | (E3188) | mg/L | - | 2.0 | 10.0 |
| Solids, Total Ignited | (E3188) | mg/L | - | 2.0 | 10.0 |
| Sulphate | (E3004) | µg/m$^3$ $SO_4$ | 28.6 | 0.1 | 0.5 |
| Sulphate | (E3013) | µg/g | 1000 | 0.5 | 2.5 |
| Sulphate | (E3172) | mg/L $SO_4$ | 100 | 0.5 | 2.5 |
| Sulphide | (E3100) | µg/L $S^{2-}$ | 100 | 2.0 | 10.0 |
| Turbidity | (E3311) | FTU | 2000 | 0.05 | 0.25 |

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